



Atom Probe Tomography & Microscopy
CONFERENCE PROGRAM
17 – 21 September 2023, Leuven (BE)



Dear colleague,

We welcome you to join – **in person** – the Atom Probe Tomography & Microscopy 2023 conference.

This 60th International Field Emission Symposium will bring together newcomers and experts from various scientific disciplines (physics, material science, geology, engineering, etc.) to discuss recent research results and future directions on aspects related to high field physics, nanoscience and atom probe microscopy.

This 5-day event will be the perfect platform to **(re)connect** with research fellows and to strengthen your scientific **network** during the workshop, informal gatherings, poster sessions and social events. The formal program will include a [J. H. Block Lecture](#) by an international renown expert in the field, invited talks, regular oral presentations, and the [prestigious Erwin Müller Outstanding Emerging Scientist Award competition](#).

The APT&M conference series is a major international forum in the field of Atom Probe Tomography & Microscopy and its applications. Topics of interest include fundamental physics; laser-matter interactions; heat transfer and optical properties at the nanoscale; reconstruction methods; modelling and computational methods; nano-scale characterization of (functional) materials and nanostructures (metals, non-metals, semiconductors, ceramics, organics, biological, liquids); sample preparation, instrumentation and technique development; field ion microscopy (FIM); correlative and combined techniques (TEM, SIMS, etc.); and more.

This will be the first in-person APT&M conference after a five-year break.

Let's be **#APTtogether** again.

Claudia Fleischmann
Conference Chair



André Vantomme
Conference Chair



Scientific Program Committee

- Oana Cojocaru-Miredin (Freiburg University, Germany)
- Claudia Fleischmann (imec & KU Leuven, Belgium) [chair]
- Baptiste Gault (MPIE, Germany)
- Richard Morris (imec, Belgium)
- Daniel Perea (PNNL, US)
- Steven Reddy (Curtin University, Australia)
- Takatori Sato (University of Sydney, Australia)
- André Vantomme (KU Leuven, Belgium) [chair]

Local Organizing Committee

- Isabelle Boesmans (KU Leuven)
- Claudia Fleischmann (IMEC and KU Leuven, Chair)
- Vitaly Krasnov (IMEC and KU Leuven)
- Richard Morris (IMEC)
- Jhao-Rong Lin (IMEC and KU Leuven)
- Jeroen Scheerder (IMEC)
- Nadya Spettel (KU Leuven)
- André Vantomme (KU Leuven, Chair)
- The JABCO-team (www.jabco.be)

Sponsors

Platinum



Gold



Silver



Bronze



General information

Date:

- 17 – 21 September 2023

Venue:

- MARIA THERESIA COLLEGE
Sint-Michielsstraat 6
3000 Leuven, Belgium

Final program is available on our website:

www.aptm2023.be

Contact: aptm2023@jabco.be



Registration

Registration and Information Desk

The Registration and Information Desk is situated in the hallway of the Maria Theresia College, Sint-Michielsstraat 6, 3000 Leuven. Please follow the signage upon arrival at the venue.

The Registration and Information Desk will be **open throughout the conference** for any questions you may have. Please do not hesitate to come to the registration desk, we are happy to help!

On Monday, September 18 it might get busy at the registration desk. To avoid long queues, we strongly advise you to pick up your badge and conference bag in time.

Registration will be open from 07:30am, while the sessions will only start at 09:00.

Conference badges

All registered participants will be given a name badge upon registration at the registration desk. This badge is your official pass to the conference venue and the social activities you registered for. We kindly request you to wear your badge visibly **on all conference occasions**.

WIFI

We offer free Wi-Fi in all university buildings. Scan the QR-code to get access. This code can be found here or on the registration desk. Once you scan the QR-code, you should be able to access the Wi-Fi network (iott). Password: **2NcnEI?F**



Scientific program

Full program

The **full program** is available on our website. For an overview of the presentations in each session, simply click on the title of the session. The abstracts can also be consulted in the **online abstract book**.

The poster sessions will take place during the poster sessions on Tuesday, September 19. These sessions will be accompanied by complementary drinks.

The World Leader in Elemental & Isotopic Microanalysis

**From Scientific
Instruments
for Research
to Metrology
Solutions for
Semiconductors**

- Geochemistry
- Cosmochemistry
- Geochronology
- Environmental Studies
- Cell & Microbiology
- Drug Discovery
- Nuclear Science
- Metals & Alloys
- Display
- Photovoltaics
- Fuel Cells
- LEDs
- Semiconductors
- Optoelectronics

...

NanoSIMS 50L



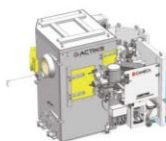
IMS 1300-HR³ | KLEORA



IMS 7f-Auto | IMS 7f-GEO



ACTINIS



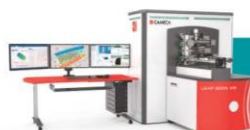
SKAPHIA



EIKOS-UV[™]



LEAP[®] 6000 XR | Invizo 6000[™]



AKONIS



IMS Wf | SC Ultra



SIMS 4550



Biography

J.H. Block Lecture



Christoph Freysoldt, PhD

Group Leader, Max-Planck-Institut für Eisenforschung *GmbH*, Düsseldorf, Germany

Pushing the limits of APT and FIM by pushing theoretical approaches

Christoph Freysoldt studied Chemistry at Leipzig university from 1997-2002, with a one-year break at Uppsala University in 2000-2001. He then moved to the Fritz Haber Institute of the Max Planck Society to pursue his PhD in Physics, which he received from the Free University Berlin in 2007. In the same year, he started at Max-Planck Institut für Eisenforschung (MPIE) in the Computational Materials Design Department to work on point defect simulations, which would become one of his major research areas. At MPIE, he was assigned scientific group leader in 2007 and head of the department IT group in 2012. In 2009, he also became responsible for the development of the department's own code for density functional theory (DFT) calculations, named SPHInX.

Christoph Freysoldt's research focuses on computing properties of point defects in semiconductors and insulators using DFT, method development in the context of ab initio electronic structure theory, the study of electrified surfaces at the atomic scale, and the use of machine learning algorithms for exploring data-rich experiments such as APT and STEM. In 2010, he received the Volker-Heine Young Investigator award for proposing a reliable correction scheme for artificial Coulomb interactions in charged-defect supercell calculations, which quickly became state of the art in the field.

Plenary Lecture



David J. Larson, PhD

Director of Marketing and Product Management for CAMECA Instruments Inc

30 Years of Atom Probe Tomography

Dr. David J. Larson is currently Director of Marketing and Product Management for CAMECA Instruments Inc.. He received his PhD. degree in Materials Science from the University of Wisconsin – Madison. He has previously held permanent or adjunct positions at Seagate Technology, Oak Ridge National Laboratory, the University of Oxford, the University of Alabama, the University of Wisconsin, and the University of Sydney. He is a Fellow of the Microscopy Society of America, an Editor of *Microscopy & Microanalysis*, and was President of the International Field Emission Society from 2014-2021. He has approximately 300 publications, holds eight patents, and may be reached at david.larson@ametec.com.

Invited speakers



Ann N. Chiamonti, PhD

Project Leader, EUV Atom Probe Tomography
Material Measurement Laboratory, NIST, USA

Atom Probe Tomography with a Wavelength-Tunable Femtosecond-Pulsed Coherent Extreme Ultraviolet Light Source

Dr. Chiamonti is currently the Material Measurement Laboratory leader of the Extreme Ultraviolet Atom Probe Project at the National Institute of Standards and Technology in Boulder, Colorado, USA. She specializes in materials characterization using electron- and ion-based imaging and scattering techniques, primarily transmission electron microscopy and atom probe tomography. Dr. Chiamonti earned degrees in Materials Science and Engineering from the University of Michigan (B.S.E.) and Northwestern University (Ph.D.). She completed a three-year postdoctoral fellowship in the Materials Science Division at Argonne National Laboratory before joining the Applied Chemicals and Materials Division of the National Institute of Standards and Technology in 2009.



Prof. Peter Felfer

Professor of materials science
Institute for General Materials Properties, Department of Materials Science, Friedrich-Alexander Universität Erlangen-Nürnberg, Germany

A fully open source toolchain for atom probe tomography of hydrogen

Peter Felfer is currently holding a professorship in atom probe tomography and 3D nanoanalytics at the Friedrich-Alexander Universität Erlangen-Nürnberg. He received his MSc. in Materials Science from the Leoben University in Austria and a PhD from the University of Sydney. His research focus for much of his career was the characterization of metallic materials, especially steels and superalloys. In recent years, after receiving an ERC starting grant for his project 'Fundamentals of Hydrogen in Structural Materials' he has been developing a research program in the area of structural materials for hydrogen applications. This includes experiments for materials characterization (APT, TDS) starting at very low hydrogen partial pressures of 10-12 mbar up to hydrogen exposure at high hydrogen pressures of 1000 bar, thus spanning 18 orders of magnitude. With his research group, he has built and developed custom made APT instrumentation including an ultra-low hydrogen APT instrument, cryo transfer equipment and a FIM. This was done together with software development for APT data acquisition, calibration/ reconstruction and 3D data analysis. All this forms an end-to-end APT toolchain that is fully open source for anyone to build their own system for custom experiments.



Natalie Holmes, PhD

Senior Research Fellow

Australian Centre for Microscopy and Microanalysis, The University of Sydney, Australia

Atom Probe Tomography for Predicting Timelines of Bioceramic Bone Scaffold Integration *in vivo*

Dr Natalie Holmes is a Senior Research Fellow at the University of Sydney where she studies the structure-function relationships in nanostructured materials with the advanced atom probe tomography suite. Her research focus is on bioceramic bone implants and biological tissues, and tracking implant integration by monitoring elemental transfer across the bone-implant interface. She obtained her PhD at the University of Newcastle in 2016, and following that led research projects at the University of Newcastle and Karlstad University (Sweden) in developing nanostructured materials using advanced synchrotron microscopy techniques, with applications in biomedical devices and solar cells.



Sebastian Koelling, PhD

Research Associate

Department of Engineering Physics, Polytechnique Montréal, Canada

Hidden structures in Atom Probe Tomography data

Dr. Sebastian Koelling is working as a research associate at Polytechnique Montreal. He earned his PhD at IMEC in 2012 working on establishing Atom Probe Tomography as a viable analysis method in monitoring semiconductor manufacturing processes. Since then, he has worked on utilizing Atom Probe for a wide range of material classes ranging from metals over superconductors to semiconducting nano-materials and biominerals. He is currently working on establishing a new Atom Probe laboratory named PolyAPT in Montreal. The first of its kind in Quebec. PolyAPT will focus on providing service for the local ecosystem and on advancing the use of APT to support the development of new bio-, battery, and quantum materials.



Ingrid McCarroll, PhD

Senior Research Associate

Max-Planck-Institut für Eisenforschung GmbH, Germany

Re-evaluating our understanding of magnesium oxidation using atom probe tomography

Dr. Ingrid McCarroll has recently taken up a position as a postdoctoral researcher at the Max Planck Institute for Iron Research. Her previous experience includes 4.5 years as a senior research assistant at The University of Sydney, where she fulfilled the role of Atom Probe Scientist. Ingrid completed her PhD with Prof. Julie Cairney at The University of Sydney using atom probe tomography to study oxidation of steels and magnesium alloys. As part of her PhD, Ingrid spent a year at The University of Oxford where she developed, in collaboration with Dr. Daniel Haley, a reaction cell attached to an atom probe. This reaction cell enabled the deuteration and oxidation of materials, with direct vacuum transfer between the reaction cell and the atom probe analysis chamber. In her role as atom probe scientist, Ingrid continued to develop methodologies for studying early oxidation of magnesium alloys and the deuteration of materials. Together with Alexander Rosenthal she implemented equipment that enabled a cryogenic and vacuum transfer integrated laboratory, connecting a FIB/SEM, atom probe, and purpose-built glovebox. Ingrid's studies include the relationship between hydrogen and the embrittlement of steels and the influence of hydrogen on the early stages of magnesium corrosion.



Isabelle Mouton, PhD

CNRS Research Engineer

University Grenoble Alpes, CNRS, Grenoble INP, SIMAP, France

Atom probe tomography of Zr-alloys using in nuclear fuel cladding

Isabelle Mouton has recently been appointed as CNRS research engineer at SIMAP laboratory in Grenoble in charge of the APT platform following the installation of a brand new LEAP6000HR. Previously she was leading research at CEA Saclay on nuclear materials with an emphasis on nanostructural investigation with APT especially on Zr alloy based cladding tubes. Isabelle has a wide expertise in APT studies. Before joining the CEA, she earned a PhD at the University of Rouen, France and completed her experience with postdoctoral positions at CEA Grenoble, France and Max-Planck institute in Dusseldorf, Germany. Since 2011, her research topics have evolved from semiconductor materials and microelectronic devices microanalysis to metallic materials, and particularly materials for the nuclear industry.



Prof. Emily Peterman

Associate Professor & Department Chair of the Earth and Oceanographic Science
Department at Bowdoin College, Maine, USA

Earthquakes and atoms: Nanoscale insights into fault evolution

Dr. Emily Peterman is a geoscientist who obtained her BA from Middlebury College and her PhD from the University of California, Santa Barbara in the USA. She pursued postdoctoral research at the University of California, Santa Cruz and Stanford University before assuming her current role as an associate professor and department chair at Bowdoin College in Maine, USA. Dr. Peterman's research program focuses on investigating the impact of micro- and nano-scale processes on crustal-scale tectonism and the evolution of lithosphere strength. Her research methodology involves combining field, laboratory, and modeling work across different spatial and temporal scales. She specializes in analyzing mineral chemistry and crystal microstructure to determine the processes, conditions, and timing of mineral (re)crystallization. Dr. Peterman has recently undertaken projects utilizing atom probe tomography to examine trace element segregations in both naturally occurring and experimentally treated minerals.



Prof. Sophie Primig

Professor of Physical Metallurgy
School of Materials Science & Engineering, UNSW Sydney, Australia

Advancements in the processing of aerospace alloys enabled by correlative microscopy

Sophie Primig is a Professor in the School of Materials Science & Engineering at UNSW Sydney. She was awarded both her MEng (2008) and PhD (2012) in Materials Science & Engineering from Montanuniversitaet Leoben, Austria. Following a short period as Post-Doctoral Researcher and an academic position at Leoben, she moved to UNSW Sydney in 2015, initially as Lecturer. She was an Australian Research Council (ARC) DECRA Fellow (2018-2020) and a UNSW Scientia Fellow (2019-2022).

Her research interests are in Physical Metallurgy. She has a track record in both fundamental and applied research. Her research goal is to develop an advanced capability in structure-property relationships across the processing routes of structural metallic materials via additive manufacturing and advanced thermo-mechanical routes. She combines state-of-the-art experimental techniques such as multiscale correlative microscopy including atom probe microscopy with mechanical testing and contemporary modelling approaches. The focus of her research is on aerospace alloys, and this has often been linked closely to the needs of industrial partners.

Sophie has attracted funding via the ARC Linkage and Discovery schemes, industry collaborations, and the Australia-US Multidisciplinary University Research Initiative Program. She is an Editor of Journal of Materials Science, Vice Chair of the TMS Phase Transformations Committee, and former President of the Materials Australia NSW Branch.



Hossein Sepehri-Amin, PhD

Group leader, Green Magnetic Materials Group, NIMS, Tsukuba, Japan
 National Institute for Materials Science, Tsukuba, Japan

**Correlative (S)TEM/APT analysis of magnetic materials
 for green energy conversions**

Dr. H. Sepehri-Amin is a materials scientist working as group leader in green magnetic materials group at National Institute for Materials Science (NIMS), Tsukuba, Japan. He is also associate professor at the University of Tsukuba. He earned his Ph. D. in material science and engineering from the University of Tsukuba in 2011. His research interest is about development of high-performance magnetic materials for green energy conversions by a combinatorial research approach of multiscale microstructure characterizations using SEM/(S)TEM/APT, micromagnetic simulations, and materials processing. He has published 148 peer reviewed journal papers (total citation of 4885, h-index:34 based on web of science) and more than 32 invited talks in the domestic and international conferences.



Prof. Mattias Thuvander

Associate Professor
 Chalmers University of Technology, Göteborg, Sweden


Atom Probe Tomography and in-situ Synchrotron Study of a Precipitation Hardened Steel

Associate Prof. Thuvander obtained his PhD degree at Chalmers University of Technology in 1998. The thesis work dealt with atom probe field ion microscopy (APFIM) of grain boundary segregation and precipitation in non-hardenable Ni-base alloys for nuclear steam generators. He did a post-doc in Oxford, working with Prof. George Smith and Prof. Alfred Cerezo using APT to study nanocrystalline Ni and Ni-Fe. After ten years in Swedish industry, he returned to Chalmers in 2009. Since then, he has been involved in materials research and the application of APT in various areas, mainly alloys for nuclear applications, but also steel, Ni-base alloys, hard thin films, implants etc. He is currently head of the Division Microstructure Physics.

Defects... buried, complex, hard to access

When failure analysis is most challenging,
we've got complete solutions for you:

- Fault Isolation
- Sample Preparation
- Imaging & Analysis

 Learn more



For research use only. Not for use in diagnostic procedures. For current certifications, visit thermofisher.com/certifications
© 2023 Thermo Fisher Scientific Inc. All rights reserved. All trademarks are the property of Thermo Fisher Scientific and its subsidiaries unless otherwise specified. AD0070-EN-08-2023

thermo scientific

FERROVAC

ULTRA HIGH VACUUM TECHNOLOGY

OUR VISION

- Connecting a multitude of complimentary preparation and analysis methods
- Connecting labs, institutes and synchrotrons across borders
- Connecting researchers through a common platform, so they can work on saving the planet

SPEAK TO US ABOUT YOUR **CONTROLLED ENVIRONMENT WORKFLOW** NEEDS BY CALLING +41 44 273 16 38 OR SENDING AN EMAIL TO SALES@FERROVAC.COM TODAY.



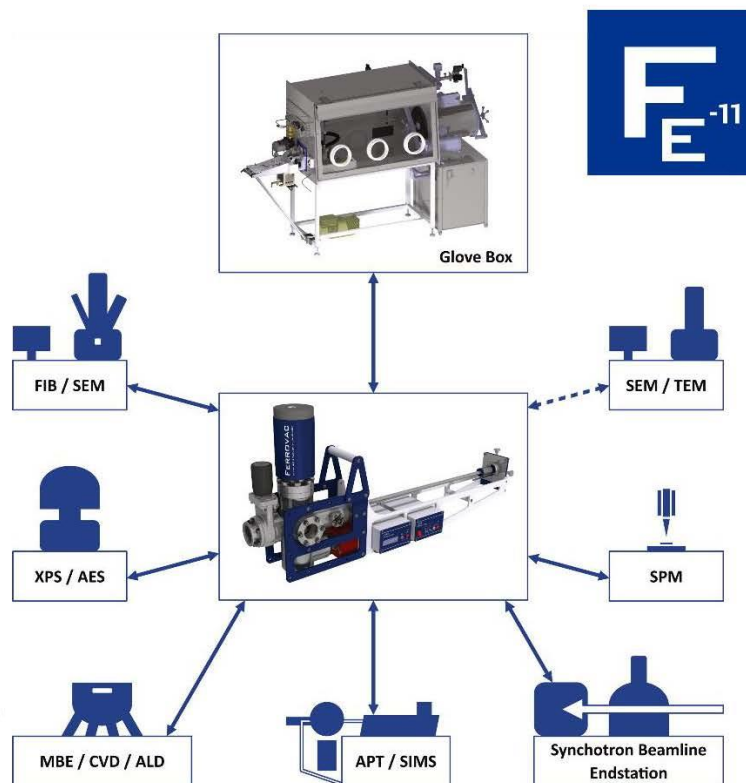
UHV Suitcase



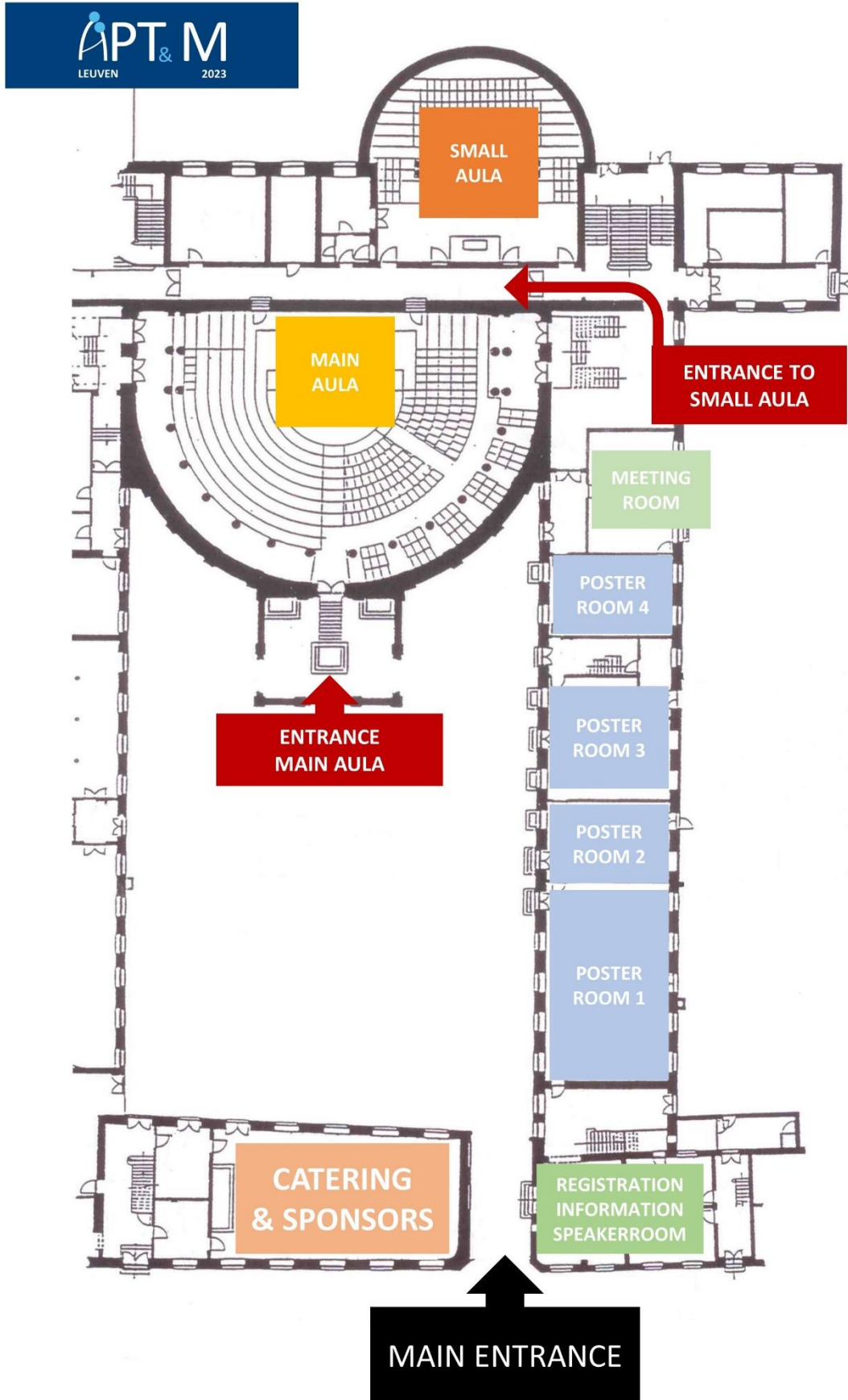
FerroLoader



Glovebox



Find your way at the venue



Schedule at a glance

Sunday, September 17 th	
9:00 – 17:00	Registration <i>Location: Entrance Hall</i>
9:30 – 10:30	ISO Standardization Kick-off Meeting (<i>open to all APT&M 2023 participants</i>) <i>Location: 0.07</i>
10:30 – 10:50	Coffee break <i>Location: 0.03</i>
10:50 – 12:00	IFES Technical Committee Meeting (<i>open to all APT&M 2023 participants</i>) <i>Location: 0.07</i>
12:00 – 13:00	Lunch break <i>(no catering provided)</i>
13:00 – 14:45	2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i>
	IFES Steering Committee Business Meeting <i>Location: 0.08</i>
14:45 – 15:15	Coffee break <i>Location: 0.03</i>
15:15 – 17:00	2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i>
	IFES Steering Committee Business Meeting <i>Location: 0.08</i>
17:00 – 18:00	Women in APT Happy Hour <i>Location: Resto Agora Leuven (co-hosted by Cameca)</i>

Monday, September 18 th	
7:30	Registration <i>Location: Entrance Hall</i>
9:00 – 9:15	Welcome to APT&M 2023 <i>Location: Main Aula</i>
9:15 – 10:00	Plenary Lecture <i>Location: Main Aula Chair: Claudia Fleischmann (imec, KU Leuven)</i>
10:00 – 11:00	E. W. Müller Award Competition (EM) <i>Location: Main Aula Chair: Arun Devaraj (PNNL)</i>
11:00 – 11:30	Coffee break
11:30 – 13:00	E. W. Müller Award Competition (EM) <i>Location: Main Aula Chair: Arun Devaraj (PNNL)</i>
13:00 – 14:15	Lunch break
14:15 – 15:45	1. Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides <i>Location: Main Aula Chair: Claudia Fleischmann (imec, KU Leuven)</i>
15:45 – 16:00	ISC Nominations <i>Location: Main Aula</i>
17:00 – 19:00	Reception <i>Location: Faculty Club</i>

Tuesday, September 19th

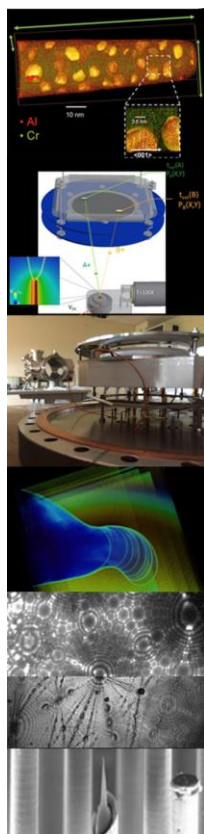
7:30	Registration <i>Location: Entrance Hall</i>	
8:45 – 9:30	J. H. Block Lecture <i>Location: Main Aula Chair: Baptiste Gault (MPIE)</i>	
9:30 – 10:15	2. Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations <i>Location: Main Aula Chair: Baptiste Gault (MPIE)</i>	
10:15 – 10:45	Coffee break	
10:45 – 12:15	3. Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation <i>Location: Main Aula Chair: Oana Cojocaru-Miredin (University of Freiburg)</i>	
12:15 – 13:30	Lunch break	
13:30 – 15:00	4. Nano-scale characterization of Metals and Alloys <i>Location: Main Aula Chair: Julie Cairney (University of Sydney)</i>	
15:00 – 15:30	Group Picture & Coffee break	
15:30 – 16:00	5. Nano-scale characterization of Nuclear and Radioactive Materials (Invited Talk) <i>Location: Main Aula Chair: Paul Bagot (University of Oxford)</i>	
16:05 – 17:05	5. Nano-scale characterization of Nuclear and Radioactive Materials <i>Location: Main Aula Chair: Paul Bagot (University of Oxford)</i>	6. Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software <i>Location: Small Aula Chair: Markus Kühbach (HU Berlin)</i>
17:05 – 19:00	Poster Session <i>Location: Ground Floor</i>	

Wednesday, September 20th

7:30	Registration <i>Location: Entrance Hall</i>	
8:00 – 08:45	IFES Steering committee - Reporting to the Community <i>Location: Main Aula François Vurpillot (Université de Rouen)</i>	
08:45 – 10:15	7. Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter <i>Location: Main Aula Chair: James Douglas (Imperial College London)</i>	
10:15 – 10:45	Coffee break	
10:45 – 12:00	8. Nano-scale characterization of Geological and Planetary Materials <i>Location: Main Aula Chair: Stephan Gerstl (ETHZ)</i>	
12:30 – 12:45	Departure bus to Brugge (Lunch Box)	

Thursday, September 21st

7:30	Registration <i>Location: Entrance Hall</i>	
9:00 – 9:30	9. Nano-scale characterization of Metals and Alloys (Invited Talk) <i>Location: Main Aula Chair: Isabelle Mouton (CNRS)</i>	
9:35 – 10:50	9. Nano-scale characterization of Metals and Alloys <i>Location: Main Aula Chair: Isabelle Mouton (CNRS)</i>	10. Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation <i>Location: Small Aula Chair: Ingrid McCarroll (MPIE)</i>
10:50 – 11:20	Coffee break	
11:20 – 11:50	11. Nano-scale characterization of Metals and Alloys (Invited Talk) <i>Location: Main Aula Chair: Takanori Sato (University of Sydney)</i>	
11:55 – 13:10	11. Nano-scale characterization of Metals and Alloys <i>Location: Main Aula Chair: Takanori Sato (University of Sydney)</i>	12. Nano-scale characterization of Semiconductor Materials and Devices <i>Location: Small Aula Chair: Andre Vantomme (KU Leuven)</i>
13:10 – 14:25	Lunch break	
14:25 – 15:25	13. Advances in Hydrogen detection <i>Location: Main Aula Chair: Steven Reddy (Curtin University)</i>	
15:25 – 16:25	14. Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software <i>Location: Main Aula Chair: Richard Morris (imec)</i>	
16:25 – 16:35	Closing remarks & Departure	
17:00	Optional Lab Tour (please register)	



15th APT School on Atom Probe Tomography Rouen November 13-17, 2023

Scope

This school will provide a thorough background on the principles of the atom probe technique, from the theory of the field evaporation to data mining. Lectures will focus on the basics of APT with a special emphasis on up-to-date techniques (laser-pulsing, DLD detectors, FIB specimen preparation). Beside this lecture series, practicals are organized including specimen preparation, Field Ion Microscopy, APT analysis and data mining.

Registration

Deadline September 30th, 2023

The participation to the school is free of charge

The number of participants is limited to ~20 people

Online registration: <https://sites.google.com/view/aptschool2023/home>

Venue

University of Rouen

Groupe de Physique des Matériaux (UMR CNRS 6634)

76801 Saint Etienne du Rouvray – France

Tel.: + 33 (0)2 32 95 50 21



Invited Lecture

Frédéric de Geuser

(SIMAP Laboratory, Grenoble, France)

Contact

Lorenzo Rigutti (lorenzo.rigutti@univ-rouen.fr)

JEOL

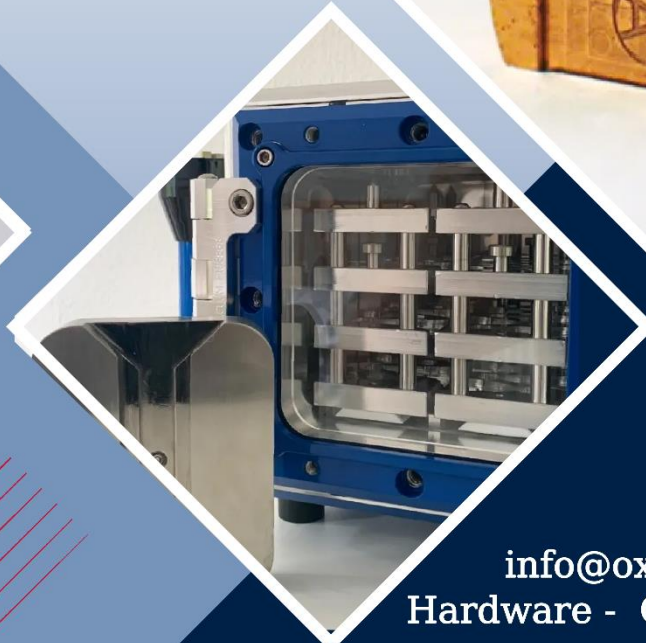
Solutions for Innovation



OXFORD ATOMIC

Microscopy Accessories

oxfordatomic.com



info@oxfordatomic.com
Hardware - Consulting - Software

Social activities

Women in APT Happy Hour

- We welcome all women attending the conference at the Women in APT Happy Hour. This drink will take place Resto Agora (Grote Markt 3, Leuven) on **Sunday, September 17 from 05:00 pm until 6:00 pm.**

The Women in APT Happy Hour is offered by



Welcome reception

- The welcome reception will take place in the Faculty Club (Groot Begijnhof 14, Leuven) on **Monday, September 18 from 05:00 pm until 7:00 pm.**

The welcome reception is partly sponsored by



Poster sessions reception

- The poster sessions will take place at the Maria Theresia College (Sint-Michielsstraat 6, Leuven) on Tuesday, **September 19 from 05:20 pm until 07:30 pm.** They will be accompanied by a reception with some drinks.

Excursion to Bruges & Conference Dinner






- The excursion to Bruges will take place on **Wednesday afternoon, September 20.**

After a guided tour and a boat trip on the canals of the idyllic city of Bruges, the evening will end with a brewery visit to De Halve Maan and a closing conference dinner.



Leuven City Map



-  Railway Station
-  Maria Theresia College
-  Women in APT Happy Hour
-  Welcome Reception
-  Bus Excursion Bruges

Detailed program

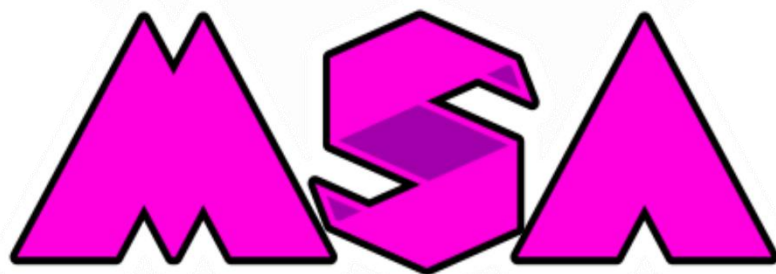
Sunday, September 17 th			
09:00 – 17:00	Registration <i>Location: Entrance Hall</i>		
09:30 – 10:30	ISO Standardization Kick-off Meeting (open to all APT&M 2023 participants) <i>Location: 0.07</i>		
10:30 – 10:50	Coffee break		
10:50 – 12:00	IFES Technical Committee Meeting (open to all APT&M 2023 participants) <i>Location: 0.07</i>		
12:00 – 13:00	Lunch break <i>(no catering provided)</i>		
13:00 – 14:45	<table border="1"> <tr> <td> 2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i> <ul style="list-style-type: none"> – CAMECA's Accessories and Consumables – Cryo Tips and Tricks – Adaptive Reconstruction Tools in AP Suite™ – Wide Field of View DUV Atom Probe </td> <td> IFES Steering Committee Business Meeting <i>Location: 0.08</i> </td> </tr> </table>	2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i> <ul style="list-style-type: none"> – CAMECA's Accessories and Consumables – Cryo Tips and Tricks – Adaptive Reconstruction Tools in AP Suite™ – Wide Field of View DUV Atom Probe 	IFES Steering Committee Business Meeting <i>Location: 0.08</i>
2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i> <ul style="list-style-type: none"> – CAMECA's Accessories and Consumables – Cryo Tips and Tricks – Adaptive Reconstruction Tools in AP Suite™ – Wide Field of View DUV Atom Probe 	IFES Steering Committee Business Meeting <i>Location: 0.08</i>		
14:45 – 15:15	Coffee break		
15:15 – 17:00	<table border="1"> <tr> <td> 2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i> <ul style="list-style-type: none"> – The imec and CAMECA Joint Development Project – AP Suite Access Program – Open Discussion on APT Topics </td> <td> IFES Steering Committee Business Meeting <i>Location: 0.08</i> </td> </tr> </table>	2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i> <ul style="list-style-type: none"> – The imec and CAMECA Joint Development Project – AP Suite Access Program – Open Discussion on APT Topics 	IFES Steering Committee Business Meeting <i>Location: 0.08</i>
2023 Cameca Atom Probe Users Meeting <i>Location: Small Aula (open to all APT&M 2023 participants)</i> <ul style="list-style-type: none"> – The imec and CAMECA Joint Development Project – AP Suite Access Program – Open Discussion on APT Topics 	IFES Steering Committee Business Meeting <i>Location: 0.08</i>		
17:00 – 18:00	Women in APT Happy Hour <i>Location: Resto Agora Leuven (co-hosted by Cameca)</i>		

Monday, September 18 th	
7:30 – 17:00	Registration Location: Entrance Hall
9:00 – 9:15	Welcome to APT&M 2023 Location: Main Aula Chair: Claudia Fleischmann (imec, KU Leuven) and André Vantomme (KU Leuven)
9:15 – 10:00	Plenary Lecture Location: Main Aula Chair: Claudia Fleischmann (imec, KU Leuven) 30 Years of Atom Probe Tomography David J. Larson (Cameca)
10:00 – 13:00	E. W. Müller Award Competition (EM) Location: Main Aula Chair: Arun Devaraj (PNNL)
10:00	EM1 Insights from atom probe tomography into stress-corrosion cracking in high-strength Al-alloys Martí López Freixes (Max-Planck-Institut für Eisenforschung GmbH)
10:30	EM2 Developing Novel Methods for Analysing Primitive Astromaterials using Atom Probe Tomography Nicole D Nevill (Lunar and Planetary Institute)
11:00 – 11:30	Coffee break
11:30	EM3 Surface Dynamics of Field Evaporation in Silicon Carbide Samba Ndiaye (CNRS-Groupe de physique des matériaux (GPM))
12:00	EM4 Application of cryo-atom probe tomography to the study of solid - liquid interfaces at near atomic scale Tim M. Schwarz (Max-Planck-Institut für Eisenforschung GmbH)
12:30	EM5 Analysis of the effect of element Y on the antioxidation behavior of cryo-milled oxide-dispersion-strengthened ferritic steel Won-Sang Shin (Inha University)
13:00 – 14:15	Lunch break (+ EM committee meeting)
14:15 – 15:45	I. Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides Location: Main Aula Chair: Claudia Fleischmann (imec, KU Leuven)
14:15	1.1 [INVITED] Correlative (S)TEM/APT analysis of magnetic materials for green energy conversions Hossein Sepehri-Amin (NIMS)
14:45	1.2 Challenges in probing the solid electrolytes of all solid-state battery by atom probe tomography Oana Cojocaru-Mirédin (University of Freiburg)
15:00	1.3 Laser Wavelength Dependence on Perovskite Interface Elemental Diffusion During Atom Probe Experiments Jonathan Poplawsky (Oak Ridge National Laboratory)
15:15	1.4 APT analysis of protective alumina scale formation in Cr ₂ AlC MAX phase Anicha Reuban (Forschungszentrum Jülich GmbH)
15:30	1.5 Diamond: a hard material to atom probe James O. Douglas (Imperial College London)
15:45 – 16:00	ISC Nominations Location: Main Aula
16:00 – 17:00	Transfer to Faculty club (Groot Begijnhof 14, 3000 Leuven - 15 min. walk from conference venue)
17:00 – 19:00	Reception Location: Faculty Club











Tuesday, September 19 th	
7:30 – 19:00	Registration Location: Entrance Hall
8:45 – 9:30	J. H. Block Lecture Location: Main Aula Chair: Baptiste Gault (MPIE) Pushing the limits of APT and FIM by pushing theoretical approaches Christoph Freysoldt (Max-Planck-Institut für Eisenforschung, Düsseldorf)
9:30 – 10:15	2. Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations Location: Main Aula Chair: Baptiste Gault (MPIE)
9:30	2.1 The dynamic atom-probe: past, presence and perspectives Norbert Kruse (Washington State University)
9:45	2.2 Determining the apex temperature of a silicon atom probe tip upon UV laser pulsing: experimental considerations Jeroen E. Scheerder (imec)
10:00	2.3 Anomalous isotope effect of field-evaporated nitrogen ions in γ' -Fe ₄ N Jun Takahashi (Nippon Steel Corporation)
10:15 – 10:45	Coffee break
10:45 – 12:15	3. Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation Location: Main Aula Chair: Oana Cojocaru-Miredin (University of Freiburg)
10:45	3.1 [INVITED] Atom Probe Tomography Using Wavelength-Tunable, Femtosecond-Pulsed Coherent Extreme Ultraviolet Radiation Ann N. Chiamonti (National Institute of Standards and Technology)
11:15	3.2 Ultra-Short Pulse Laser Assisted Atom Probe Mehrpapad Monajem (Friedrich-Alexander-Universität Erlangen-Nürnberg)
11:30	3.3 Evaluating LEAP® 6000 DUV Interactions and Survivability Ty J. Prosa (CAMECA)
11:45	3.4 Automated site-specific 3DAP tip preparation by using script-controllable FIB-SEM system Jun Uzuhashi (Washington State University)
12:00	3.5 Development of the TOMO Microscope Joseph Bunton (CAMECA)
12:15 – 13:30	Lunch break
13:30 – 15:00	4. Nano-scale characterization of Metals and Alloys Location: Main Aula Chair: Julie Cairney (University of Sydney)
13:30	4.1 [INVITED] Microstructural engineering via metal additive manufacturing of stainless steels enabled by atom probe microscopy Sophie Primig (The University of Sydney)
14:00	4.2 Grain boundary segregation analysis in martensitic steel grades using atom probe tomography Davit Melkonyan (OCAS N.V.)
14:15	4.3 Characterization of intermetallic and carbide nano-particles in a novel dual precipitation strengthening martensitic steel Ze Sheng (KTH Royal Institute of Technology)
14:30	4.4 Correlative microscopic analysis of nano-scale precipitate evolution and localization in Ni alloyed Fe-Mn-Al-C steel K. G. Pradeep (IIT Madras)
14:45	4.5 APT study of corrosion products on a weathering steel exposed in SO ₂ -rich industrial atmosphere Xiaohuan Liu (Nanjing University of Science and Technology)
15:00 – 15:30	Group Picture & Coffee break

Tuesday, September 19 th (cont.)		
15:30 – 16:00	5. Nano-scale characterization of Nuclear and Radioactive Materials <i>Location: Main Aula Chair: Paul Bagot (University of Oxford)</i>	
15:30	5.1 [INVITED] Atom probe tomography of Zr-alloys using in nuclear fuel cladding <i>Isabelle Mouton (University Grenoble Alpes, CNRS)</i>	
16:00 – 16:05	Transfer	
16:05 – 17:05	5. Nano-scale characterization of Nuclear and Radioactive Materials <i>Location: Main Aula Chair: Paul Bagot (University of Oxford)</i>	6. Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software <i>Location: Small Aula Chair: Markus Kühbach (HU Berlin)</i>
16:05	5.2 Using APT and TEM to Uncover the Mechanism(s) of Accelerated Corrosion of Zirconium Alloys in High-Lithium Environments Calum Cunningham (National Nuclear Laboratory)	6.1 Development of a repository for APT grain boundary excess data Alexander Reichmann (Montanuniversität Leoben)
16:20	5.3 Atom probe tomography of zirconium fuel cladding after boiling water reactor operation David Mayweg (Chalmers University of Technology)	6.2 Extracting thermodynamics information from local composition fluctuations in solids: Extended theory and its application to simulated and experimental atom probe data Jianshu Zheng (University of Stuttgart)
16:35	5.4 Atom probe tomography to investigate irradiation damage in tungsten for nuclear fusion applications Kieran C. Rivers (University of Oxford)	6.3 Towards large-scale field evaporation simulations with ab initio accuracy Shyam Katnagallu (Max-Planck-Institut für Eisenforschung, Düsseldorf)
16:50	5.5 Synergetic effects of Mn, Ni, and Si on the formation of Mn-Ni-Si clusters and irradiation hardening in reactor pressure vessel model steels Deepak Sharma (Université de Rouen)	6.4 Compensating imaging distortions in a commercial reflectron type atom probe Peter Felfer (Friedrich-Alexander Universität Erlangen-Nürnberg)
17:05 – 19:05	Poster Session <i>Location: Ground Floor</i>	

microscopysuppliesaustralia.com



MICROSCOPY SUPPLIES AUSTRALIA

 Electropolished PNTY™ Grids: Pack of 10 \$550	 Loading Block \$100	 Specimen Holder Mk II \$250	 APT/SEM Sample Rack \$29	 Vacuum Storage for 10 APT Holders \$650
 Specimen Holder Mk I \$200	 Atom Probe Kit: A \$1,000	 Atom Probe Kit: C \$3,000	 Loading Kit D \$325	 Loading Kit E \$550

Wednesday, September 20 th	
7:30 – 12:00	Registration Location: Entrance Hall
8:00 – 08:45	IFES Steering committee - Reporting to the Community Location: Main Aula François Vurpillot (Université de Rouen)
08:45 – 10:15	7. Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter Location: Main Aula Chair: James Douglas (Imperial College London)
8:45	7.1 [INVITED] Atom Probe Tomography for Predicting Timelines of Bioceramic Bone Scaffold Integration in vivo Natalie Holmes (The University of Sydney)
9:15	7.2 Advancements in 3D Nanoscale Characterization of Organic-Mineral Interfaces in Dental Enamel Jack Grimm (Pacific Northwest National Laboratory)
9:30	7.3 Towards Near-Native State 3D Analysis of Rehydrated E. coli Bacterial Cells using Atom Probe Tomography Florant Exeriter (Deakin University)
9:45	7.4 Site-specific preparation and atom probe tomography of bulk water ice and amino acid solutions Eric V. Woods (Max-Planck-Institut für Eisenforschung)
10:00	7.5 Cryo Atom Probe Tomography Studies of 5CB, 8CB Liquid Crystals Kuan Meng (University of Stuttgart)
10:15 – 10:45	Coffee break
10:45 – 12:00	8. Nano-scale characterization of Geological and Planetary Materials Location: Main Aula Chair: Stephan Gerstl (ETHZ)
10:45	8.1 [INVITED] Earthquakes and atoms: Nanoscale insights into fault evolution Emily M. Peterman (Bowdoin College)
11:15	8.2 A nanoscale vision of primitive meteorites: APT characterization of the fine-grained mesostasis Rick Verberne (University of Copenhagen)
11:30	8.3 Nanoscale Constraints on the Early-Earth Geodynamo Steven M. Reddy (Curtin University)
11:45	8.4 Containment of Radionuclide Contaminants in Mineral Interfaces David Saxey (Curtin University)
12:00 – 12:30	Transfer to bus station in groups (Ferdinand Smoldersplein)
12:30	Departure bus to Brugge (lunch box provided)

Thursday, September 21 st		
7:30	Registration Location: Entrance Hall	
9:00 – 9:30	9. Nano-scale characterization of Metals and Alloys Location: Main Aula Chair: Isabelle Mouton (CNRS)	
9:30	9.1 [INVITED] Atom probe tomography and in-situ synchrotron study of a precipitation hardened steel <i>Mattias Thuvander (Chalmers University of Technology)</i>	
9:30-9:35	Transfer	
9:35 – 10:50	9. Nano-scale characterization of Metals and Alloys Location: Main Aula Chair: Isabelle Mouton (CNRS)	10. Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation Location: Small Aula Chair: Ingrid McCarroll (MPIE)
9:35	9.2 Mapping the influence of dendritic segregation on the nano-scale in single crystal Ni-based superalloys <i>Victoria C. I. Strutt (University of Oxford)</i>	10.1 Analysis of ice-loaded nanoporous copper tips using atom probe tomography <i>Levi Tegg (The University of Sydney)</i>
9:50	9.3 Exploring avenues for tailoring the thermal stability of hierarchical microstructures in Ni-based superalloys via phase targeted alloy design <i>Florian Vogel (Hainan University)</i>	10.2 Cryo-Atom Probe Tomography to investigate frozen liquids in templated materials <i>Oliver Waszkiewicz (University of Oxford)</i>
10:05	9.4 A thorough study of grain boundary segregation, energy, and width in copper–nickel thin films using Atom Probe Tomography <i>Sebastian Eich (University of Stuttgart)</i>	10.3 Deep UV-laser assisted atom probe tomography analysis of glass materials for protein encapsulation <i>Gustav Eriksson (University of Oxford)</i>
10:20	9.5 Nanoscale characterisation of powder processed Ni ODS alloys <i>Megan Carter (University of Oxford)</i>	10.4 Correlative in-situ Liquid Cell Electrochemistry TEM and Cryogenic APT of Liquid-Solid Interfaces <i>Shelly Conroy (Imperial College London)</i>
10:35	9.6 Morphological and chemical evolution of nanoscale precipitates in Alloy 718 as a function of processing <i>Vitor Vieira Rielli (The University of Sydney)</i>	10.5 A Reactor Chamber Attached to the Atom Probe Tomography for Enabling In Situ Studies of Solute Partitioning to Interfaces During Heat Treatments and Oxidation <i>Arun Devaraj (Pacific Northwest National Laboratory)</i>
10:50 – 11:20	Coffee break	
11:20 – 11:50	11. Nano-scale characterization of Metals and Alloys Location: Main Aula Chair: Takanori Sato (University of Sydney)	
11:20	11.1 [INVITED] Re-evaluating our understanding of magnesium oxidation using atom probe tomography <i>Ingrid McCarroll (Max-Planck-Institut für Eisenforschung Duesseldorf)</i>	
11:50 – 11:55	Transfer	
11:55 – 13:10	11. Nano-scale characterization of Metals and Alloys Location: Main Aula Chair: Takanori Sato (University of Sydney)	12. Nano-scale characterization of Semiconductor Materials and Devices Location: Small Aula Chair: Andre Vantomme (KU Leuven)
11:55	11.2 High-temperature oxidation of alloys: APT insights into nanoscale mechanisms <i>Ivan Povstugar (Central Institute for Engineering Juelich)</i>	12.1 Photonic Atom Probe study of Sub-Bandgap Luminescence in a GaN/InGaN heterostructure <i>Lorenzo Rigutti (Université de Rouen)</i>
12:10	11.3 High-temperature oxidation at Nb-Al ₂ O ₃ interfaces at the nanoscale <i>Torben Boll (Karlsruhe Institute of Technology)</i>	12.2 Exploring Stacking Faults in GaN-Based LEDs through APT Investigation <i>Ruiying Shu (University of Oxford)</i>
12:25	11.4 Revising nanostructure and magnetism of Sm(Co,Zr) _{7.5} magnets <i>Nikita Polin (Max-Planck-Institut für Eisenforschung GmbH)</i>	12.3 Extreme Ultraviolet Pulsed Atom Probe Tomography of β -Ga ₂ O ₃ <i>Luis Miaja-Avila (National Institute of Standards and Technology)</i>
12:40	11.5 Carbon controlled growth of columnar ferrite nanostructures in iron carbon thin films <i>Jonas Arlt (University of Göttingen)</i>	12.4 A field dependent study on the compositional analysis of boron doped silicon using a GRID and normal local electrode. <i>Richard J. H. Morris (imec)</i>
12:55	11.6 Probing Dealloying Mechanisms in Thin-Film Nanoporous Metals Using Atom Probe Tomography <i>Ezgi Hatipoglu (Max-Planck-Institut für Eisenforschung GmbH)</i>	12.5 Quantitative Dopant Analysis in Silicon via Atom Probe Tomography <i>Karen DeRocher (National Institute of Standards and Technology)</i>
13:10 – 14:25	Lunch break	

Thursday, September 21 st (cont.)	
14:25 – 15:25	13. Advances in Hydrogen detection <i>Location: Main Aula Chair: Steven Reddy (Curtin University)</i>
14:25	13.1 [INVITED] A fully open source toolchain for atom probe tomography of hydrogen Peter Felfer (Friedrich-Alexander Universität Erlangen-Nürnberg)
14:55	13.2 Hydrogen trapping capability of Hybrid steel investigated by deuterium charged samples in atom probe experiments Severin Jakob (Chalmers University of Technology)
15:10	13.3 Visualizing radiation-induced damage by Hydrogen/Deuterium Aparna Saksena (Max-Planck-Institut für Eisenforschung GmbH)
15:25 – 16:25	14. Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software <i>Location: Main Aula Chair: Richard Morris (imec)</i>
15:25	14.1 [INVITED] Hidden structures in Atom Probe Tomography data Sebastian Koelling (Polytechnique Montreal)
15:55	14.2 An Investigation into Probabilistically Assigning Peak-Overlapped Ions When Performing Cluster Searches in APT Data Benjamin Jenkins (Université de Rouen)
16:10	14.3 Visualizing radiation-induced damage by Hydrogen/Deuterium Yue Li (Max-Planck-Institut für Eisenforschung GmbH)
16:25 – 16:35	Closing remarks & Departure <i>Location: Main Aula Chair: Claudia Fleischmann (imec, KU Leuven) and André Vantomme (KU Leuven)</i>
17:00	Optional Lab Tour (please register) Imec cleanroom Leuven NanoCentre KU Leuven Ion- and Molecular Beam Laboratory



Vacuum Technology for Space Research on Earth

Benefit from our **complete range** of turbopumps as well as backing pumps, leak detectors, components, measurement and analysis equipment as well as vacuum chambers **for simulating space conditions**. Get to know our products: the magnetically levitated **turbopumps** from the ATH series, **vacuum chambers** for highly demanding application or the innovative **Roots pumps** of the HiLobe series.



Plenary Lecture

30 YEARS OF ATOM PROBE TOMOGRAPHY

David J. Larson¹

¹*CAMECA Instruments, Inc.*

The field of atom probe tomography has seen tremendous technological advances in the past three decades. Thirty years ago, nobody prepared specimens using focused ion beam milling, with its combination of liftout and site specific capabilities. Very few researchers had a laser atom probe. The concept of a “local” electrode, and the many advantages that it conveys, did not exist. A brief perusal of the proceedings of the 39th International Field Emission Symposium organized by Prof. H. J. Kreuzer and held in Halifax in 1992 (published in 1993) confirms these statements [1]. This lecture will present, from a rather personal perspective, the evolution of some of these concepts along with the contributions of some of the researchers who made possible the current world of atom probe tomography.

[1] H.Jürgen Kreuzer & M. K. Miller Appl. Surf. Sci. 67 (1993)

Erwin Müller Competition

EM1

INSIGHTS FROM ATOM PROBE TOMOGRAPHY INTO STRESS-CORROSION CRACKING IN HIGH-STRENGTH AL-ALLOYS

Martí López Freixes¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

The high-strength 7xxx series aluminium alloys, Al-Zn-Mg-(Cu), can fulfil the need for light, high-strength materials necessary to reduce carbon-emissions from vehicles but they can be sensitive to stress-corrosion cracking (SCC) depending on temper, loading and environmental conditions. SCC crack growth in these alloys has primarily been associated to hydrogen embrittlement (HE), as H is generated as a by-product of oxidation at the crack tip. Here, we performed industry standard crack growth tests and then analyzed the stress-corrosion cracks by atom probe tomography (APT), which has a very high chemical and spatial resolution. H generated at the crack tip is found segregated at dislocations and grain boundaries ahead of the crack, indicative of hydrogen-enhanced localized plasticity (HELP) and hydrogen-enhanced decohesion (HEDE). We also report on hydroxide formation at the crack tip, rich in the main alloying elements Zn, Mg and Cu, irrespective of the cracking environment. Evidence of dissolution of the strengthening precipitates at and ahead of the crack is observed, along with solute diffusion processes involving the grain boundary (GB). APT has enabled the discovery of invaluable phenomena thanks to its unmatched capabilities, with implications for future high-strength alloy designs key to achieving more environmentally resistant materials.

Erwin Müller Competition

EM2

DEVELOPING NOVEL METHODS FOR ANALYSING PRIMITIVE ASTROMATERIALS USING ATOM PROBE TOMOGRAPHY

Nicole D Nevill¹, Phil A Bland², David W Saxey³, William D. A. Rickard³, Md Zakaria Quadir³, Steven Reddy², Nick E Timms², Lucy V Forman², Luke Daly⁴

¹Lunar and Planetary Institute, USA

²Space Science and Technology Centre, Curtin University, Australia

³John de Laeter Centre, Curtin University, Australia

⁴University of Glasgow, United Kingdom

Primitive carbonaceous comets and asteroids preserve the fundamental building blocks of our solar system. Interplanetary Dust Particles (IDPs) sample a more diverse range of carbonaceous bodies than meteorites, their components of which are considered among the most informative materials regarding early Solar System and presolar processes [1]. However, IDPs are porous, multiphase assemblages with fragile microstructures comprised of hydrous, anhydrous, organic and inorganic nanophases, many which reach the spatial resolution limits of currently applied techniques. Here we present an affective approach for studying IDPs using atom probe tomography (APT) and demonstrate its value as a quantitative tool for IDP analysis. To address porosity, IDPs were embedded in epoxy. To improve structural integrity, needles were redesigned with larger shank angles (7 – 12°) and a 600 – 800 nm thick base. Testing various acquisition parameters identified parameters constrained during test runs of pure epoxy needles improved data quality and acquisition stability, regardless of the phases embedded within the specimen. Results demonstrated i) APT can be an effective complementary technique for detailed study of microstructural and geochemical signatures in IDPs, essential for advancing our understanding of the formation of ours and external planetary systems. ii) N functional groups between carbonaceous regions were more diverse than first thought, suggesting formation in isolated environments. iii) Monoclinic tridymite nanospheres, consistent with low-temperature hydrothermal formation on the parent body, were dehydrated, indicating exposure to a thermal heating event within the solar nebula. iv) APT can measure salt crystals, with results showing Na-rich halite with minor K and trace Mg, Si and Al, phases which have historically proved challenging to measure even at qualitative levels [2]. v) We recently demonstrated the application and value of studying O-rich presolar grains in APT [3]. Using the latter, we demonstrate the importance of coordinating the approach presented within this study with a newly developed FIB technique [4] for studying astromaterials and improving targeting capabilities.

[1] J. P. Bradley, Meteorites and cosmochemical processes, 1 (2014), 181–213. [2] Zolensky et al, Philos. Trans. Royal Soc. A, 375 (2017). [3] Nevill et al, LPSC, 54 (2023), 1707 [4] Nevill et al, Goldschmidt, (2023), 15328.

Erwin Müller Competition

EM3

SURFACE DYNAMICS OF FIELD EVAPORATION IN SILICON CARBIDE

Samba Ndiaye¹, C. Bacchi¹, B. Klaes¹, M. Canino², F. Vurpillot¹, L. Rigutti¹

¹*University of Rouen Normandie, France*

²*CNR-IMM, Italy*

Atom Probe Tomography (APT) of compound semiconductors may present specific biases in the determination of chemical compositions because of the different behavior of different elements with respect to field evaporation [1], [2]. Silicon carbide (SiC) may be considered as a model system for the study of field ion evaporation of carbides, which must be understood in order to obtain accurate analyses of these systems by atom probe tomography (APT). As for other wide bandgap semiconductors, the measurement of composition of SiC by APT presents biases depending on the experimental parameters. Unlike silicon, carbon is characterized by a complex surface behavior, including formation of molecules and the tendency to produce correlated evaporation. Furthermore, the spatial precision of 3D reconstructions is strongly degraded in the direction parallel to the sample surface, which points out to a strong roughening or dynamic degradation of the surface. This is confirmed by field ion microscopy (FIM) analysis which reveals that atoms may move on the sample surface under the influence of the high electric field. This complex surface behavior eventually translates into hidden detection events and, therefore, to errors in the measurement of composition.

Erwin Müller Competition

EM4

APPLICATION OF CRYO-ATOM PROBE TOMOGRAPHY TO THE STUDY OF SOLID - LIQUID INTERFACES AT NEAR ATOMIC SCALE

Tim M. Schwarz¹, Eric Woods¹, Leonardo S. Aota¹, Xuyang (Rhett) Zhou¹, Ingrid McCarroll¹, Baptist Gault¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

APT measurements require a needle-shaped specimen with an end radius in the range of below 100 nm, which, for frozen liquids, represents significant challenges that have only recently been overcome.

My work aims to address some of the remaining analytical challenges for APT with the analysis of (frozen) liquids, biomolecules, including advancing the understanding of their fragmentation and field evaporation behavior, and enable analysis of interfaces between solids and liquids.

In my talk, I will discuss my studies on frozen liquids with increasing complexity and their evaporation behavior and towards liquid-solid interfaces that control for instance corrosion, wet chemical synthesis, or catalytic processes. To advance the understanding of these processes at interfaces, there is a need to facilitate the analysis on the smallest length scale, only accessible by APT. Therefore the mechanisms of early-stage corrosion in a Mg-alloy used as an application-related example for implants, was investigated.

A defined corrosion state is preserved by plunging the sample into liquid nitrogen and APT specimens are prepared with a cryo-FIB and subsequently analyzed with APT. This example demonstrated how cryo-APT reveals intermediate corrosion products, namely a hydroxide, partitioning of alloying elements between metal, hydroxide, and oxide etc. This new trove of novel information was not previously accessible, hindering the development of a predictive model of the aqueous corrosion of these alloys.

Erwin Müller Competition

EM5

ANALYSIS OF THE EFFECT OF ELEMENT Y ON THE ANTIOXIDATION BEHAVIOR OF CRYO-MILLED OXIDE-DISPERSION-STRENGTHENED FERRITIC STEEL

Won-Sang Shin¹, Sung-Il Baik², David N. Seidman², Kee-Ahn Lee¹, Changkyoo Park³, Yoon-Jun Kim¹

¹*Inha University, South-Korea*

²*Northwestern University Center for Atom-Probe Tomography (NUCAPT), USA*

³*Seoul National University of Science and Technology, South-Korea*

Oxide-dispersion-strengthened (ODS) steel exhibits excellent creep and oxidation resistance, and therefore is used in severe environments such as nuclear reactors. Its nanometer-sized oxide features and the use of a combination of appropriate factors and parameters, such as milling methods, temperature, and time, for its processing enable the steel to withstand high-temperature degradation. Y-Ti-O nanometer-sized particles (NPs) with a diameter of less than 5 nm play a key role in the properties of these materials. Therefore, the oxidation resistance characteristics of ODS steel manufactured by ball milling at two temperatures were analyzed through various techniques via Atom-Probe Tomography (APT). ODS Steel were manufactured by ball milling with Y₂O₃ powder at two temperatures, room temperature and cryotemperature (≈ -150 °C). Both samples were exposed to air for 6000 h at 800 °C, and after long-term exposure both samples showed subsurface degradation occurred concurrently such as internal precipitation, phase formation, and phase dissolution. The resulting oxide scaling kinetics were more sluggish for cryotemperature compared to room temperature condition. Construction of isoconcentration surfaces of the top most oxide layer in cryotemperature sample reveals Y layer formed at the boundary between substrate and oxide layer suggesting Y atoms reacted first with oxygen to form Y₂O₃. On the basis of the reduction of the interfacial energies calculated by using the Gibbsian interfacial excess and the partial radial distribution functions of major alloying elements, Y atoms in room temperature condition were found to show a strong tendency to form Y-Ti-O nanoparticles, while N atoms did not form NPs on the surface. On the other hand, N atoms at cryotemperature condition participated in the formation of Y-Ti-O(N) NPs, and was prone to transformed into TiN precipitates to reduce the interfacial energy as the oxidation process progressed. The transformation from Y-Ti-O(N) to TiN precipitate drove Y atoms to be released from NPs and segregated at the matrix/oxide interface. Segregated Y atoms effectively prevented further oxidation of the matrix, and thus oxide scaling kinetics became slower than solutioned in Y-Ti-O NPs.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

1.1

CORRELATIVE (S)TEM/APT ANALYSIS OF MAGNETIC MATERIALS FOR GREEN ENERGY CONVERSIONS

Hossein Sepehri-Amin¹, T. T. Sasaki¹, T. Ohkubo¹, K. Hono¹

¹NIMS, Tsukuba, Japan

Magnetic materials have a wide range of applications from energy conversion to data storage. The functionality of magnetic materials in general can be defined by their magnetic/thermal hysteresis which is strongly micro/nanostructure dependent. In this presentation, we will show examples of how correlative electron microscopy and atom probe tomography analysis have led to the development of desired large hysteresis in the permanent magnets and minimization of hysteresis for soft magnets and magnetocaloric materials [1-5]. In the case of permanent magnets, (Nd,Dy)-Fe-B based magnets are currently the material of choice for traction motors of hybrid/electric vehicles and generators of wind turbines. However, the scarcity of Dy has urged researchers to eliminate its use. Dy-free Nd-Fe-B magnets have low resistance against magnetization reversal (coercivity) and therefore cannot store energy, especially at elevated temperatures. Our detailed correlative (S)TEM/APT analysis has shown that the existence of a thin intergranular phase with a high content of ferromagnetic elements is responsible for the low coercivity of Dy-free Nd-Fe-B magnets. Based on this knowledge, we have successfully modified the composition/structure of the intergranular phases and interphase interfaces in the permanent magnets leading to the development of high coercivity and thermally stable Dy-free Nd-Fe-B based permanent magnets [4]. We will also discuss how the engineering of secondary phases and the elimination of inhomogeneities can benefit the minimization of hysteresis in magnetocaloric materials for efficient magnetic refrigeration. As an example, we will show how the elimination of nanoscale compositional fluctuations in Ni-Mn-In based Heusler alloys leads to a uniform martensite/austenite phase transition and thus minimizes thermal hysteresis, which is beneficial for the reversibility of the magnetocaloric effect [5].

[1] H. Sepehri-Amin et al. *Acta Mater.* 126 (2017) 1.

[2] A. Bolyachkin, H. Sepehri-Amin, et al. *Acta Mater.* 227 (2022) 117716.

[3] T. T. Sasaki, H. Sepehri-Amin et al. *MRS Bulletin* 47 (2022) 688.

[4] M. Korent, et al. *Scripta Mater.* 205 (2021) 114207.

[5] H. Sepehri-Amin et al. *Acta Mater.* 147 (2018) 342.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

1.2

CHALLENGES IN PROBING THE SOLID ELECTROLYTES OF ALL SOLID-STATE BATTERY BY ATOM PROBE TOMOGRAPHY

Oana Cojocaru-Mirédin¹, Jan Köttgen², Frank Tietz³

¹*University of Freiburg, Germany*

²*RWTH Aachen, Germany*

³*Forschungszentrum Jülich GmbH, Germany*

All solid-state battery (ASSB) is one of the most important energy devices for future green energy, but its performance and reliability have to be further improved. The ASSB contains three main parts such as the anode, cathode and solid electrolyte. Extensive research has been done in the last years on developing a dendrite-free solid electrolyte. Yet, the plated light alkalis (such as Lithium or Sodium) reacts irreversibly with the solid electrolyte and forms insoluble dendrites very deep down inside the electrolyte. This leads to an increase of the stress to accommodate the volume increase explaining why the electrolytes are generally very brittle and difficult to be analyzed by atom probe tomography (APT). Moreover, this stress can open cracks and, hence, lead to loss of electrical contact at the anode/solid electrolyte interface (interfacial delamination). Therefore, in the present work the challenges in investigating the solid electrolytes such as $\text{Li}_6\text{.6La}_3\text{Zr}_{1.6}\text{O}_{12}$ and $\text{Na}_{1+x}\text{Zr}_2\text{SixP}_3\text{-xO}_{12}$ will be presented. Moreover, possible ways will be provided of how to measure these brittle and highly resistive solid electrolytes by correlative microscopy[1]. In conclusion, this work opens the possibility to investigate solid electrolytes down to the atomic level and extends the applicability of the APT to these very brittle and resistive battery materials.

[1] Cojocaru-Mirédin, O., et al., Journal of Power Sources, 2022. 539: p. 231417.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

1.3

LASER WAVELENGTH DEPENDENCE ON PEROVSKITE INTERFACE ELEMENTAL DIFFUSION DURING ATOM PROBE EXPERIMENTS

Jonathan Poplawsky¹, Jan Köttgen², Frank Tietz³

¹*Oak Ridge National Laboratory, USA*

SrTiO₃ (STO)/LaMnO₃ (LMO) perovskite metal oxide superstructures are highly desired for spintronic application because they show enhanced magnetism compared to bulk LMO. SrTiO₃(STO)/LaMnO₃(LMO)/La(Al,Sc)O₃(LASO) superstructures with different growth orders (STO→LMO→LASO→STO...(STO→LMO sample) and STO→LASO→LMO→STO...(LMO→STO sample) were grown by atomic layer deposition (ALD) to assess the effect of the LMO/STO termination plane on the magnetic properties. STEM-annual bright field (ABF) imaging and electron energy loss spectroscopy (EELS) didn't show noticeable differences between the samples; however, STEM-EELS data is difficult to interpret because of channeling effects and it is only sensitive to approximately 1 at. %. Therefore, atom probe tomography (APT) was used to assess chemical diffusion across the interfaces to complement the STEM results. The initial APT experiments were performed using a LEAP4000XHR equipped with a 3.5 eV laser. Sample yield was less than 10%, and only small volumes were collected. The results show Mn diffusion into STO and Ti diffusion into LMO for the LMO→STO sample (sample with worse magnetic properties). This diffusion was not found in the STO→LMO sample. The LMO→STO sample was also run using a LEAP6000XR equipped with a 4.8 eV laser. Three full superstructures were captured out of three attempts (100% yield) and much larger volumes were collected. Surprisingly, the results only show Mn diffusion into STO for the LMO→STO sample, which is inconsistent with the Ti diffusion found within the LEAP4000XHR data. Repeat LEAP4000XHR experiments using backside sample preparation indicate Ti retention during the APT experiment creates an artificial Ti diffusion profile into LMO. Ti retention for only the LEAP4000XHR experiments can be explained by STO's band structure even though 3.5 eV and 4.8 eV excitations are larger than STO's band gap. Overall, the 3.5 eV laser excites an indirect band transition, while the 4.8 eV laser excites a direct band transition. The results reveal that field evaporation may be dependent on direct band gap excitation in large band gap materials. Mechanisms for differences in field evaporation behavior for STO/LMO/LASO superstructures subject to 3.5 and 4.8 eV laser excitations will be discussed.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

1.4

APT ANALYSIS OF PROTECTIVE ALUMINA SCALE FORMATION IN CR₂AlC MAX PHASE

Anicha Reuban¹, Ivan Povstugar¹, Marcin Rasinski¹, Andrey Litnovsky¹, Christian Linsmeier¹, Jesus Gonzalez-Julian²

¹Forschungszentrum Jülich GmbH, Germany

²RWTH Aachen University, Germany

In a Concentrated Solar Power (CSP) plant, solar thermal energy is used to generate electricity by focusing sunlight onto a receiver. To increase efficiency, the receiver must withstand temperatures greater than 800 °C, be resistant to oxidation by air and/or corrosion by molten salts and maintain its properties over time. MAX phases, which are ternary carbides and nitrides with a unique combination of ceramic and metallic properties, are candidate materials for the receiver. In this work, the high-temperature oxidation of the alumina-forming MAX phase Chromium Aluminum Carbide (Cr₂AlC) is studied. Unlike typical alumina-forming alloy systems, Cr₂AlC does not form a pronounced depletion zone below the oxide, but forms a discrete, continuous layer of chromium carbide (Cr₇C₃) instead. Hence, the mechanism of oxidation in Cr₂AlC is different from classical alloys. To understand the oxidation mechanism, the elemental distribution across grain boundaries and interfaces of various regions of the oxide scale, carbide and bulk Cr₂AlC are analyzed with Atom Probe Tomography (APT), since these are pathways for the diffusion of aluminum and oxygen ions to form the oxide layer. It is observed that the diffusion of aluminum proceeds differently through the Cr₂AlC and Cr₇C₃ layer, controlling the supply of aluminum ions at different stages of oxide formation. From the analysis of the bulk composition it is also seen that Cr₂AlC can only tolerate up to ~1.5 at.% of aluminum depletion before transforming into Cr₇C₃, showing that Cr₂AlC is a highly stoichiometric compound. The effect of dissolved chromium in the alumina scale on the ionic transport, and segregation of impurities such as iron and silicon to interfaces were also addressed. Based on these observations, a possible mechanism for the alumina scale formation is proposed.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

1.5

DIAMOND: A HARD MATERIAL TO ATOM PROBE

James O. Douglas¹, Paul May², Michael Moody³, Tomas Martin²

¹*Imperial College London, United Kingdom*

²*University of Bristol, United Kingdom*

³*University of Oxford, United Kingdom*

Diamond is often found at the top of the league table of material properties – it has exceptional thermal conductivity, physical and radiation hardness, optical transparency and a wide band gap of 5.5eV, allowing a variety of dopants. These properties make it an attractive material for heat sinks high power devices, quantum photon sources (via nitrogen-vacancy centres) and radiation shielding. Unfortunately, many of the properties that make diamond such an exciting material also make it challenging to study using atom probe tomography. Traditional focused ion beam (FIB) methods of sample preparation typically results in preferential etching of any defects or grain boundaries in the material, causing columning and curtaining. When analysing diamond in the laser-assisted atom probe, other challenges appear – such as its optical transparency and thermal conductivity, which make it challenging to add sufficient laser power for consistent evaporation, resulting in high electric fields and poor specimen yield. The high evaporation field requirement of diamond also can lead to migration and preferential evaporation of gallium introduced through FIB sample preparation. There are some examples in the literature where diamond has been analysed using atom probe, showing an interface between C¹² and C¹³ chemically vapor deposited diamond [1] and diamond extrasolar particles in metal matrix [2]. However, when using the high laser energies successfully used on such homogenous specimens on doped diamond, the thermal effects causes diffusion of dopants resulting in non-physical clustering. Advances in specimen preparation via plasma focused ion beam with additional gas species such as O and Ar can mitigate some of these issues. In this study, a series of chemically vapor deposited, boron-doped multicrystalline diamond is analysed using a series of different sample preparation and analysis steps, in order to understand how best to study the atomic-scale chemistry of this important but challenging material.

[1] S Mukherjee et al, Laser-Assisted Field Evaporation and Three-Dimensional Atom-by-Atom Mapping of Diamond Isotopic Homojunctions, *Nano Lett.* 16 (2016)

[2] Heck et al, Atom-probe analyses of nanodiamond from Allende, *Meteoritics & Planetary Science* 49 (2014)

J.H. Block Lecture

PUSHING THE LIMITS OF APT AND FIM BY PUSHING THEORETICAL APPROACHES

Christoph Freysoldt¹, Shalini Bhatt¹, Shyam Katnagallu¹, Alaukik Saxena¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

Field ion microscopy (FIM) and atom probe tomography (APT) share a decade-long history and have provided numerous unique insights into surface processes and elemental distributions down to the atomic scale. Yet, the elementary process of field-induced ionization and evaporation, that underpins these techniques, is far from being fully understood, notably for the chemically diverse samples that practitioners deal with on a daily basis. In practice, those complicated details are largely ignored since the data evaluation algorithms anyway rely on drastic simplifications to make them manageable. Unfortunately, this opens the door to ill-controlled artifacts, distortions, and uncertainties. Progress in developing a quantitative theory is therefore not only of academic interest, but should help in misinterpreting experimental observations, in both routine applications and cutting-edge method development. Obviously, ionization and bond-breaking cannot be assessed without taking the electronic structure into account. For this, today's method of choice is density-functional theory (DFT). I will show a few examples how we use DFT to overcome some of the previous limitations in theoretical models by replacing assumptions by quantitative predictions that account for the specific electronic structure effects at hand. On the one hand, our investigations of thermally driven field evaporation highlight the role of surface configurations and alloying effects for the field-barrier dependence of key steps such as roll-over, surface diffusion, and bond-breaking. On the other hand, DFT-based tunneling probability maps obtained by adopting the Tersoff-Hamann theory of the scanning tunneling microscope (STM) yield, for the first time, an assessment of chemical contrast in FIM. Of course, the atomic-scale (few atoms) perspective captures only a tiny part of what APT and FIM offer. At the opposite extreme, the huge amount of data from each single experiment (let alone the series of experiments possible with modern instrumentation) is both a challenge and a largely unexploited opportunity for data-centric analysis. I will sketch some of our steps to leverage machine-learning approaches to identify and quantify chemically distinct phases, their spatial patterns, and their geometrical characteristics in a more automatic way.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

2.1

THE DYNAMIC ATOM-PROBE: PAST, PRESENCE AND PERSPECTIVES

Norbert Kruse¹, Thierry Visart de Bocarmé²,

¹*Washington State University, USA*

²*Université libre de Bruxelles, Belgium*

The past two decades have witnessed 3D-Atom Probe Tomography (APT) to develop into a highly sophisticated experimental technique for characterizing a diversity of materials and their surfaces. When it comes to identifying the local chemical composition with nano- or sub-nanoscale spatial resolution, 3D-APT may provide unprecedented insight even into highly complex material compositions. Unfortunately, every APT materials analysis is performed under UHV, i.e. ~ 12 orders of magnitude (or even more) below atmospheric pressure. This limitation scotches any attempt of studying dynamic processes affording the presence of environmental gas or liquid phase conditions. The present communication will provide an account of past attempts to overcome this problem and to perform combined field ion microscopy (FIM) and 1D-Atom Probe experiments when subjecting metallic samples to gas pressures up to 10⁻³ mbar. The data highlight the prospects of providing valuable insight into catalytic and electrocatalytic phenomena during the ongoing reaction processes. Finally, we will discuss what it would take to redesign state-of-the-art 3D-APT instrumentation to make it compatible with the presence of reduced pressures of the same order of magnitude as in 1D experiments. The dynamic 1D-Atom Probe as originally developed at the Fritz-Haber Institute of the Max Planck Society uses bell-shaped field pulses (~ 100 ns half width) with varying frequencies between 100 kHz and 1 Hz so as to allow monitoring the kinetics of surface reactions while continuously dosing the sample with reactive gaseous species. Different from 3D-Atom Probe applications, pulse fractions of 100% can be adjusted.. This allows the kinetics of nitric oxide (NO) adsorption on the surface of a Pt specimen to be studied. Next, we will inspect how arbitrary steady electric fields in addition to pulsed fields influence the adsorption and reaction behavior of NO molecules. Switching to FIM conditions in the absence of field pulses and while co-dosing NO and H₂ to a Pt specimen, spatiotemporal pattern formation due to the catalytic production of water occurs as demonstrated using video techniques. We finish the presentation by discussing the prospects of a dynamic 3D Atom-Probe enabling large field-of-view analyses of reaction processes like those mentioned above for the 1D-design.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

2.2

DETERMINING THE APEX TEMPERATURE OF A SILICON ATOM PROBE TIP UPON UV LASER PULSING: EXPERIMENTAL CONSIDERATIONS

Jeroen E. Scheerder¹, Viktor de Ridder², Egor Khramov¹, Yuan Tu¹, Richard J.H. Morris¹, Masoud Dialameh¹, André Vantomme², Claudia Fleischmann¹²

¹IMEC, Belgium

²KU Leuven, Belgium

In laser-assisted atom probe tomography (L-APT), the dominant mechanism for triggering field evaporation is believed to be a combined effect of an intense electric field and thermal pulsing. Knowledge about the underlying physical mechanisms underpinning the evaporation process remains vague, especially for semiconductors and dielectrics [1]. Furthermore, it is cumbersome to determine the exact temperature at the tip apex during laser pulsing, albeit a too high temperature could trigger atom (surface) diffusion thereby deteriorating the spatial resolution [2] and/or altering the material during analysis. This work assesses experimentally the temperature increase in a semiconductor (i.e. silicon) during L-APT ($\lambda=355$ nm, LEAP 5000XR), for a broad laser energy range (1-50 pJ). As the specimens are fabricated through lithographic techniques (pre-sharpened microtips), we leverage the quasi-identical tip geometries, and circumvent ion beam-induced defects at the specimen surface. The apex temperature is quantified through the laser induced reduction of the applied voltage (and/or the electric field) to the specimen with respect to a reference temperature [3]. Here, we demonstrate how specific experimental factors could likewise alter the voltage (electric field) irrespective of the temperature. If not properly accounted for, this reduces the accuracy and precision of the determined apex temperature. For instance, a variation in the specimen-local electrode distance of a few micrometers can alter the voltage by a similar amount as a temperature change of ~ 50 K. We show that using the size of the signal of the position-sensitive detector, one can precisely reproduce the specimen-local electrode distance between acquisitions, or even correct for any misalignment afterwards. A non-linear temperature increase with laser energy is found, opposite to the linear trend found for metals, both in literature and own data. We compare these results on Si in UV to earlier studies, in relation to the material systems, APT tools, and laser [1,3,4].

[1] T.F. Kelly et al., *Curr. Opin. Solid State Mater. Sci.* 19, 81 (2014) and references therein

[2] A. Cerezo et al., *Appl. Phys. Lett.* 88, 154103 (2006)

[3] T.F. Kellogg, *J.Appl.Phys.* 52, 5320 (1981), E. Marquis and B. Gault, *J.Appl.Phys.* 104, 08914 (2008)

[4] A. Kumar et al., *J.Appl.Phys.* 124, 245105 (2018)

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

2.3

ANOMALOUS ISOTOPE EFFECT OF FIELD-EVAPORATED NITROGEN IONS IN γ' -Fe₄N

Jun Takahashi¹, Kazuto Kawakami¹, Koyo Miura³, Mitsuhiro Hirano³, Naofumi Ohtsu³,

¹*Nippon Steel Corporation, Japan*

²*Nippon Steel Technology, Japan*

³*Kitami Institute of Technology, Japan*

When mass interference (peak-overlap in mass spectrum) occurs between field-evaporated ions in atom probe tomography (APT) analysis, the elemental concentration is generally estimated using isotope ions without mass interference based on the natural abundance. To analyze the composition of carbides, a correction method using the minor isotope (¹³C) has been proposed in order to reduce the impact of the detection-loss of multi-hit carbon ions in the delay-line detector [1]. These methods using isotope ions assume that there is no difference in field-evaporated ion species between isotope species.

The performance of APT in the quantitative analysis of nitrogen in steel has remained unclear owing to the mass interference of ¹⁴N₂⁺ and ⁵⁶Fe²⁺ at 28 Da [2]. By using the iron nitride of γ' -Fe₄¹⁵N that is produced by plasma nitriding with ¹⁵N₂ isotope gas, we revealed that the presence of missing nitrogen, likely due to neutral nitrogen or nitrogen gas molecules in field evaporation, prevents the quantitative analysis of nitrogen in current APT instruments [3]. In the experiments, we found an anomalous “isotope effect” in the generation of field-evaporated nitrogen ions (N⁺) and iron nitride ions (FeN²⁺). The generation rate of FeN²⁺ in γ' -Fe₄¹⁴N was considerably higher than that in γ' -Fe₄¹⁵N, while that of N⁺ in γ' -Fe₄¹⁴N was lower than that in γ' -Fe₄¹⁵N. The generation rates of N⁺ and FeN²⁺ were uniquely determined by the isotope ratio of ¹⁴N and ¹⁵N. This indicates that the ion species depend on the isotope species of the nitrogen atom at the kink site on the tip surface. We estimated the total energy of the nearest cut bonds for the kink of Fe and N in the γ' -Fe₄N crystal and proposed a mechanism explaining the isotope effect in field evaporation.

Such an isotope effect in field-evaporated ion species suggests that the isotope ratio of each ion species is not necessarily the same as the natural abundance. The use of isotopes should be done with great caution in APT analysis.

[1] J. Angseryd *et al.*: Ultramicroscopy 111 (2011) 609-614.

[2] W. Sha *et al.*: Surf. Sci. 266 (1992) 416-423.

[3] J. Takahashi *et al.*: Microanal. 28 (2022) 45-52.

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

3.1

ATOM PROBE TOMOGRAPHY USING WAVELENGTH-TUNABLE, FEMTOSECOND-PULSED COHERENT EXTREME ULTRAVIOLET RADIATION

Ann N. Chiaramonti¹

¹*National Institute of Standards and Technology, USA*

As the National Metrology Institute of the United States, the mission of the National Institute of Standards and Technology (NIST) is to promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology. From establishing standards for fire hose couplings to promoting cutting-edge metrology research in areas of advanced communication, quantum science, forensics, biomaterials, and even standards for peanut butter [1], NIST has always focused research efforts and measurement services in addressing contemporary societal needs. This talk will focus on new approaches to atom probe metrology for materials science.

I will discuss a world-first research program dedicated to using extreme ultraviolet (EUV) light to perform 3D atom probe tomography (APT). Ionizing radiation in the EUV region of the electromagnetic spectrum ($E_{\text{photon}} \approx 10 \text{ eV}$ to 100 eV , $\lambda = 120 \text{ nm}$ to 10 nm) is highly absorbed by any material, and offers potential new ionization and desorption pathways for APT that are not available using near-ultraviolet (NUV; $E_{\text{photon}} \approx 3.5 \text{ eV}$, $\lambda = 355 \text{ nm}$) nor the recently introduced deep-ultraviolet ($E_{\text{photon}} \approx 4.8 \text{ eV}$, $\lambda = 257.5 \text{ nm}$) light sources. The photon energies currently accessible in the NIST EUV APT system exceed the work function of any solid and ionization potential of any atom, opening the possibility of photoexcitation-based field ion emission mechanisms.

Instrument design and selected experimental results from the EUV atom probe microscope at NIST will be presented. This instrument uses wavelength-tunable femtosecond-pulsed coherent EUV radiation from phase-matched high harmonic generation in a capillary waveguide. I will show data from a variety of materials systems that demonstrate successful EUV-triggered field ion emission. Time-independent background levels, delayed evaporation tails, peak widths, charge state ratios, multiple hit counts, measured stoichiometry, and the relative number of cluster ions will be compared with NUV experiments. Some of the myriad possible ionization and desorption mechanisms present or enabled by EUV APT will be discussed.

[1] Montgomery, R. (2022), SRM NIST Standard Reference Materials Catalog January 2022, https://tsapps.nist.gov/publication/get_pdf.cfm?pub_id=934102 (Accessed May 1, 2023)

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

3.2

ULTRA-SHORT PULSE LASER ASSISTED ATOM PROBE

Mehrpada Monajem¹, Peter Felfer¹

¹*Friedrich-Alexander Universität, Erlangen-Nürnberg, Germany*

After the introduction of modern short (< 100 ps) pulse lasers into Atom Probe Tomography (APT), it has gained widespread adoption across diverse domains due to the ability to produce high-quality data from non-conductive specimens. Up to now, conventional lasers assisted atom probes have pulse duration and wavelength down to 355 nm and 15 ps respectively. However, to date, heating from the laser beam is the mechanism triggering field evaporation. Several groups around the world are working on systems with lasers promising athermal responses, including THz pulsing [1] and extreme-UV sources [2]. In field electron emission, few-cycle pulses have been successfully used to create athermal field emission [3]. While ions are arguably much heavier than electrons, it is still interesting if very short (ca. 10 fs) pulses offer improved field evaporation behavior for APT. In this talk, we show results from experiments on a field electron spectrometer, operated as a laser-assisted atom probe with 12 fs, 1550 nm laser pulses. By employing such short pulses, we can obtain a very high-resolution mass spectrum when measuring tungsten samples at room temperature. We conducted this experiment using PyCCAPT [4], our open-source Python-based package designed to manage and calibrate both the APT experiment and obtained data.

[1] Vella, Angela, et al. "High-resolution terahertz-driven atom probe tomography." *Science Advances* 7 (2021): eabd7259.

[2] Miaja-Avila, Luis, et al. "Extreme Ultraviolet Radiation Pulsed Atom Probe Tomography of III-Nitride Semiconductor Materials." *The Journal of Physical Chemistry C* 125.4 (2021): 2626-2635.

[3] Paschen, Timo, et al. "Ultrafast Strong-Field Electron Emission and Collective Effects at a One-Dimensional Nanostructure." *ACS Photonics* 2 (2023): 447-455.

[4] Mehra Monajem & Harsh Pandey. (2023). mmonajem/pyccapt: pre-release (pre-release). Zenodo. <https://doi.org/10.5281/zenodo.7588996>

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

3.3

EVALUATING LEAP® 6000 DUV INTERACTIONS AND SURVIVABILITY

Ty J. Prosa¹, Isabelle Marin¹, David A. Reinhard¹, David J. Larson¹

¹CAMECA Instruments, Inc.

The deep ultra-violet (DUV, 257.5 nm) wavelength laser, available in the LEAP and INVIZO® 6000 products, modifies the interaction between laser and specimen. Initial reports have suggested that for Si/SiO₂ interfaces, a more similar effective evaporation field results relative to 355 nm [2]. In this presentation, we will show that under DUV illumination, survivability can be achieved over a wider range of analysis conditions, allowing one to better maintain adequate survivability without sacrificing data quality. High survivability (>80%) is possible under high-field, low-LPE acquisitions (relative to 355 nm) and with higher detection rates, enabling improved reconstruction and faster time-to-knowledge. In addition, we report on acquisition for this material. This new capability can allow high quality analysis at even lower laser-pulse fraction/higher evaporation field while maintaining modest background rates (out-of-time evaporation). This study will also highlight the use of

[1] T.J. Prosa et al., *Microsc. Microanal.* 21 (2015) p.849.

[2] Ty Prosa et al., *Microsc. Microanal.* 27 (2021) p.1262.

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

3.4

AUTOMATED SITE-SPECIFIC 3DAP TIP PREPARATION BY USING SCRIPT-CONTROLLABLE FIB-SEM SYSTEM

Jun Uzuhashi¹, Tadakatsu Ohkubo¹, Kazuhiro Hono¹

¹*National Institute for Materials Science (NIMS), Japan*

The development of the atom probe specimen (tip) preparation method using a focused ion beam (FIB) with scanning electron microscopy (SEM) dual-beam system has contributed to a significant expansion of the application area of the three-dimensional atom probe (3DAP). While the recent commercial 3DAP instruments allow precise control of analysis conditions, the analytical results including mass resolution and analyzed volume size are strongly influenced by the tip shapes [1-3]. However, the tip shapes are still highly dependent on the skill of FIB-SEM operators. In recent years, FIB-SEM systems that can be automatically controlled by a scripting language have been commercialized [4,5]. In this work, we established the method to automatically fabricate 3DAP tips with a specified tip curvature and a taper angle with higher reproducibility and accuracy by using a script-controllable FIB-SEM system [3]. In addition to automatic tip shape control, “site-specific” tip preparation is now also possible. Some experimental demonstrations of the automatic tip preparation method are presented.

[1] L. Arnoldi, A. Vella, J. Houard, B. Deconihout, *Appl. Phys. Lett.* 101, 153101 (2012).

[2] A. Cerezo, P.H. Clifton, A. Gombert, G.D.W. Smith, *Ultramicroscopy* 107, 720-725 (2007).

[3] J. Uzuhashi, T. Ohkubo, K. Hono, *Ultramicroscopy* 247, 113704 (2023).

[4] B.V. Leer, R. Geurts, R. Scharfschwerdt, H. Cheng, L. Li, and R. Imlau, *Microscopy Today* 26, 18–25 (2018).

[5] M. Dutka and A. Prokhotseva, *Microscopy and Microanalysis* 25, 554-555 (2019).

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

3.5

DEVELOPMENT OF THE TOMO MICROSCOPE

Joseph Bunton¹, Russell Manzke¹, Jeff Shepard¹, Nick Brewer¹, Daniel Lenz¹, Gard Groth¹, Jesse Robinson¹, Michael Holman¹, Timothy Kaltenbach¹, Jesse Olson¹, Laurent Rousselet¹, Hugo van Leeuwen², Pleun Dona², Hans Persoon², Casper Smit², Ron van den Boogaard², Jos Hoogeveen², Nestor Hernandez Rodriguez², Marcin Gramza², Naren Hoovinakatte², Hans van Lin²

¹CAMECA Instruments, Inc.

²ThermoFisher Scientific, The Netherlands

Atom probe tomography (APT) data reconstruction becomes tractable if the shape of the atom probe emitter can be independently determined via transmission electron microscopy (TEM) [1]. Likewise, correlative studies have demonstrated the combined power of APT and TEM when analyzing microstructures ranging from advanced metals to semiconductor devices [2,3]. The complementary utility of these techniques, as well as the desire to interleave APT and TEM data, naturally led to the proposal of a combined APT-TEM instrument [4]. The TOMO microscope is currently under development as a collaboration between Thermo-Fisher Scientific™ and CAMECA®. This revolutionary instrument will be installed at the Ernst Ruska-Centre of the Julich Forschungszentrum Institute in 2024, combining for the first time in-situ high-performance TEM imaging capability and atom probe tomography. The atom probe module builds on many aspects of established CAMECA APT technology including ultraviolet laser illumination and ion optics based on the Invizo 6000® which provide a wide field of view and high mass resolving power. The design allows rapid alternation between TEM/STEM/EELS imaging and atom probe analysis. A prototype of the TOMO microscope including the fully integrated atom probe module has been completed this spring at Thermo-Fisher Scientific in Eindhoven. Performance of the APT module has been validated demonstrating performance meeting specifications. We look forward to the delivery of the fully functional instrument to FZ Julich next year.

[1] Haley, D., Petersen, T., Ringer, S.P. and Smith, G.D.W. (2011), Atom probe trajectory mapping using experimental tip shape measurements. *Journal of Microscopy*, 244: 170-180.

[2] Ceri A. Williams, Emmanuelle A. Marquis, Alfred Cerezo, George D.W. Smith (2010), Nanoscale characterisation of ODS–Eurofer 97 steel: An atom-probe tomography study. *Journal of Nuclear Materials*, 400-1: 37-45.

[3] Lauhon, Lincoln J., Praneet Adusumilli, Paul Ronsheim, Philip L. Flaitz, and Dan Lawrence (2009), Atom-Probe Tomography of Semiconductor Materials and Device Structures. *MRS Bulletin* 34.10: 738-743.

[4] Michael K. Miller, Thomas. F. Kelly, Krishna Rajan, Simon P. Ringer (2012), The Future of Atom Probe Tomography. *Materials Today*. 15-4: 158-165.

Nano-scale characterization of Metals and Alloys

4.1

MICROSTRUCTURAL ENGINEERING VIA METAL ADDITIVE MANUFACTURING OF STAINLESS STEELS ENABLED BY ATOM PROBE MICROSCOPY

Sophie Primig¹

¹*The University of Sydney, Australia*

The inherent inhomogeneity and cyclic thermal loading in metal additive manufacturing (AM) significantly change the microstructural and solid/solid interface evolution when compared to subtractive processing methods. However, the same AM characteristics may enable targeted microstructure and property control once the current fundamental understanding of the underlying mechanisms has been extended, and this is often informed by microscopy data.

In my own recent research as part of the AUSMURI project, we have made exciting discoveries which will enable tuning of microstructures and solid-solid interfaces for property control directly during 3D printing of stainless steels, without complex forming and thermal treatments in metallurgical plants.

This presentation will showcase handpicked recent results on microstructural and interface engineering via laser powder bed fusion AM of selected stainless steels for defence, aerospace, and other high-performance applications. These efforts have been enabled via multiscale correlative microscopy and atom probe microscopy (APM).

In 17-4 PH stainless steel, APM has provided valuable insights into the evolution of the matrix phase (delta-ferrite, austenite, or martensite) during printing and post-AM heat treatments, the potential for Cu clustering and precipitation, and the site-specific microstructure evolution as a function of powder chemistry and scan strategy [1,2]. In 2205 duplex stainless steel, new insights into intergranular precipitation and chemical fluctuations in the non-equilibrium as-printed delta-ferrite microstructures have been revealed via APM. Site-specific APM has shown the decoration of various types of delta-ferrite/austenite interfaces with solutes and unlocked new knowledge on nucleation and growth phenomena [3,4].

[1] M. Moyle, N. Haghdadi, X. Z. Liao, S. P. Ringer, S. Primig: *Journal of Materials Science & Technology* 117 (2022) 183-195.

[2] M. S. Moyle, N. Haghdadi, W. J. Davids, X. Z. Liao, S. P. Ringer, S. Primig: *Scripta Materialia* 219 (2022) 114896.

[3] N. Haghdadi, H. Chen, Z. Chen, S. S. Babu, X. Z. Liao, S. P. Ringer, S. Primig: *Scripta Materialia* 219 (2022) 114894.

[4] N. Haghdadi, C. Ledermueller, H. Chen, Z. Chen, Q. Liu, X. Li, G. S. Rohrer, X. Z. Liao, S. P. Ringer, S. Primig: *Materials Science & Engineering A* 835 (2022) 142695.

Nano-scale characterization of Metals and Alloys

4.2

GRAIN BOUNDARY SEGREGATION ANALYSIS IN MARTENSITIC STEEL GRADES USING ATOM PROBE TOMOGRAPHY

Davit Melkonyan¹, Koenraad Theuwissen¹, Lode Duprez¹

¹OCAS NV, Belgium

Solute segregation around grain and phase boundaries can significantly affect the macroscopic properties of steels. Understanding the mechanisms and linking the macroscopic properties to the nanoscale impurity segregation around grain boundaries can help to develop steel grades for specific applications. An important part of such an exercise consists in accurate measurements of segregated impurities with high spatial resolution and chemical sensitivity. Atom probe tomography (APT), due to its unique 3D nature, high chemical and spatial resolution, was already proven very useful for this. In this contribution, six martensitic concepts with different alloying and process strategies were investigated by means of APT to monitor the segregation at the prior austenite grain boundaries (PAGBs). The first three materials, with three specific compositions and thermal history, had a full martensitic structure. For the fourth composition, three different heat treatments were applied to obtain a martensitic matrix containing retained austenite, with different microstructure and segregation states. For each material, two APT samples were analysed to increase the statistical relevance. The main segregated elements detected around almost all the measured PAGBs are B, C, Mo, and P. As expected both the bulk composition of the analysed grades and their processing routes impact the PAGB chemistry. A detailed discussion of these impacts and possible mechanisms driving the segregation process will be presented.

Nano-scale characterization of Metals and Alloys

4.3

CHARACTERIZATION OF INTERMETALLIC AND CARBIDE NANO-PARTICLES IN A NOVEL DUAL PRECIPITATION STRENGTHENING MARTENSITIC STEEL

Ze Sheng¹, Alexander Dahlström¹, Manon Bonvalet Rolland^{1,2}, Wangzhong Mu¹, Peter Hedström¹

¹*KTH Royal Institute of Technology, Sweden*

²*UMET, Université de Lille, CNRS, France.*

By combining the precipitation-strengthening mechanisms from traditional tool steels and maraging stainless steels the company Ovako has developed a novel hybrid steel grade for bearing applications, Hybrid 60[®]. The new steel has excellent stable mechanical properties at elevated temperatures due to the formation of small secondary carbides and intermetallic particles. However, the complicated precipitation of several potential particles and their interaction needs to be investigated in detail to be able to optimize the microstructure and properties of the steel. This is the purpose of the present work. The integrated computational materials engineering (ICME) framework was used to directly tailor mechanical properties through microstructural design driven by the complex thermal treatment. Where NiAl, Ni₃Al, M₂₃C₆, M₇C₃, and M₆C have the potential to form during a set of key experiments. A Quenching/Deformation Dilatometer, Linseis L78 RITA, was used to accurately control the heating, annealing, and cooling process of the samples. The effect of slow (0.2 °C/s) and fast (50 °C/s) cooling rates after the austenitization on the subsequent precipitation during tempering was investigated using atom probe tomography (APT). The APT experiments were performed using various equipment such as LEAP 3000 XHR, LEAP 4000 HR, and EikOS-UV. The precipitate's size, composition, and number density from APT are used as validation to simulations using Langer-Schwartz-Kampmann-Wagner mean-field precipitation modelling.

Nano-scale characterization of Metals and Alloys

4.4

CORRELATIVE MICROSCOPIC ANALYSIS OF NANO-SCALE PRECIPITATE EVOLUTION AND LOCALIZATION IN NI ALLOYED FE-MN-AL-C STEEL

K. G. Pradeep¹

¹IIT Madras, India

The presence of Al in Fe-Mn-C based multi-component steels induces precipitate formation whose size, number density and distribution can be varied depending on the thermomechanical processing conditions. Further alloying of such steels with Ni promotes the formation of Ni-Al type B2 precipitates depending on the Mn content. This work therefore attempts to analyze correlatively the structure of nano-scale precipitates formed in a duplex (i.e. FCC matrix and embedded BCC precipitates) steel along with their precise local chemical composition in a high statistical manner. To achieve the above, a custom designed correlative microscopy platform was utilized in a Scanning Electron Microscope enabling imaging in transmission geometry combined with transmission Kikuchi diffraction for structure analysis while the precise local chemical composition at near atomic-resolution was obtained from the same investigated volume using Atom Probe Tomography. This correlative methodology applied to hot rolled and annealed conditions, provided deeper insights into the role of starting microstructures on the precipitation kinetics and hence on the final mechanical properties of the steel.

Nano-scale characterization of Metals and Alloys

4.5

APT STUDY OF CORROSION PRODUCTS ON A WEATHERING STEEL EXPOSED IN SO₂-RICH INDUSTRIAL ATMOSPHERE

Xiaohuan Liu¹, Gang Sha¹

¹*Nanjing University of Science and Technology, China*

Understanding corrosion mechanism of weathering steel (WS) is important for safe operation and minimizing economic loss of some key engineering structures. It is generally believed that the corrosion resistance of a WS depends on the structure and property of its inner rust layer (RL). The good corrosion resistance of WS in SO₂-polluted industrial atmosphere is attributed to the formation of non-cracked, uniform, and protective amorphous hydroxylated iron oxide. However, our understanding about the amorphous substance is limited largely due to technical challenge in the detection and study of the substances. The elemental distribution characteristics and formation mechanisms of amorphous substances in the RL has yet to be revealed. The lack of deep knowledge on the amorphous substances hindered our understanding about atmospheric corrosion of WS [1] [2]. Atom probe tomography (APT) is powerful in revealing atomistic chemical information of materials in three-dimensional. With the help of focused ion beam (FIB) technique, APT sample can be prepared at specific locations in the RL of WS. In this study, the RL on the WS after 12 months exposure in SO₂-polluted industrial atmosphere was found, consisting of an outer RL, an inner RL with fine grains, and a transition layer with amorphous phase. The fine grain layer in the inner RL was found to contain Cr- and Si-rich particles. Interestingly, for the first time, Ni elements was found to be enriched in the amorphous substance of the transition layer (Fig. 1). In-depth structural and compositional analysis has provided a deep understanding about RL formation mechanism. Deciphering the elemental distribution characteristics of RL in WS is an important step in designing structural materials with enhanced corrosion resistance.

[1] Ph. Dillmann, et al. Corrosion Science 46 (2004) 1401-1429.

[2] M. Morcillo, et al. Corrosion Science 83 (2014) 6-31.

Nano-scale characterization of Nuclear and Radioactive Materials

5.1

ATOM PROBE TOMOGRAPHY OF ZR-ALLOYS USING IN NUCLEAR FUEL CLADDING

Isabelle Mouton^{1,2,3}, Amelie Souksavat², Sylvie Doriot², Fabien Onimus², Jean Christophe Brachet², Yanhong Chang³, Siyang Wang⁴, Ben Britton⁴, Baptiste Gault³

¹*Université Grenoble Alpes, France*

²*Université Paris-Saclay, France*

³*Max-Planck-Institut für Eisenforschung GmbH, Germany.*

⁴*Imperial College London, United Kingdom*

Zirconium-based alloys are used for nuclear fuel cladding and act as the first containment barrier of the fission product tubes because of their low thermal neutron absorption cross section and their good mechanical properties. During in-service operation in water reactors, the Zr-based cladding tube is subject simultaneously to oxidation, hydrogen pick-up and to neutron irradiation. These phenomena have consequences on the microstructure and affect the alloy properties during its service lifetime.

This presentation will give an overview of different microstructure evolutions that can occur on Zr-alloys and particularly by investigating the distribution of several alloying elements by atom probe tomography.

Firstly, we will focus on the influence of the hydrogen pick-up coming either from the metal oxidation that reduces water and liberates atomic hydrogen or directly from the fuel. A consequence of this increase in hydrogen concentration is the precipitation of zirconium hydrides and these hydrides are known to be mechanically brittle. To deepen our understanding of the formation mechanism of hydrides in this system, we investigate by atom probe tomography different samples as Zr-Sn alloys exhibiting large grains microstructure and hydrogen/deuterium charged with particular attention to the grain boundary.

In addition to this study, a technological solution to decrease the Zr corrosion, both in nominal and hypothetical accidental conditions at high temperature will be presented. Indeed, it has been shown that the deposition of a thin Cr coatings on Zr-based nuclear fuel claddings provide a significant reduction in the oxidation-induced embrittlement of the nuclear fuel cladding, especially upon accidental conditions (and therefore decrease the associated gaseous hydrogen production). To guarantee the successful adhesive strength of the Cr coatings, the interface between Cr and Zr has to be finely characterized via atom probe tomography.

In the last part, evolution of the microstructure and the micro-chemistry in the Zr-Nb alloy under irradiation will be presented. The results will focus on the chemical investigation of the Zr-matrix, beta-Nb precipitates, Laves phases, radiation-enhanced precipitates, solute dissolution from the precipitates into the matrix or potential segregation related to <c>-component loops. These APT observations are correlated with TEM results obtained on the same materials in order to give a complete overview of the microstructural evolution under irradiation.

Nano-scale characterization of Nuclear and Radioactive Materials

5.2

USING APT AND TEM TO UNCOVER THE MECHANISM(S) OF ACCELERATED CORROSION OF ZIRCONIUM ALLOYS IN HIGH-LITHIUM ENVIRONMENTS

Calum Cunningham¹, Paul Styman¹, Jonathan Hawes¹, Alex Cackett¹, Alistair Garner¹

¹National Nuclear Laboratory, United Kingdom

The addition of LiOH to the coolant of pressurised water reactors (PWRs) has been commonplace for decades to control pH levels in reactor cores. It is widely known, however, that high concentrations of Li can lead to accelerated corrosion of zirconium alloy fuel cladding [1]. Li may concentrate locally where coolant flow is restricted, or wick boiling occurs – increasing the risk of premature fuel failure. To mitigate this, it is important to understand the mechanism by which Li accelerates Zircaloy oxidation, but no single hypothesis is generally agreed upon. Most proposed mechanisms require Li to be located at particular microstructural features, e.g. Li may alter the grain boundary properties of the oxide to increase the rate of oxidation. In this work, atom probe tomography (APT) has been employed to investigate the location of Li within the oxides of Zircaloy-4 samples oxidised in various high-lithium PWR-type autoclaves (ranging 60-250 wt.ppm Li and 310-350°C). APT revealed that in all cases Li segregates to oxide grain boundaries, along with Fe and H (identified by OH⁺ ions). However, the concentration of Li at grain boundaries as a function of corrosion temperature and water Li content does not align with the observed effects of these two factors on the accelerated rate of corrosion. Our observations imply that Li at grain boundaries may not be the primary driver for accelerated oxidation rates, in contradiction to several hypothesised mechanisms. In addition, transmission electron microscopy (TEM) has been used in conjunction with APT to evaluate the microstructure of accelerated zirconium oxides. Using techniques such as HAADF-STEM (high-angle annular dark field scanning TEM) and through-focus imaging, TEM has revealed the presence of both intragranular and grain boundary porosity, with some pores under a nanometre in diameter. It is possible that an interconnected network of porosity and cracks within the oxide allows for ingress of Li which in turn accelerates oxide growth. In this presentation, the results of APT and TEM studies will be presented and used to assess the various proposed mechanisms of Li-induced acceleration of Zircaloy corrosion.

[1] McDonald, Sabol & Sheppard, Zirconium in the Nuclear Industry (1984).

Nano-scale characterization of Nuclear and Radioactive Materials

5.3

ATOM PROBE TOMOGRAPHY OF ZIRCONIUM FUEL CLADDING AFTER BOILING WATER REACTOR OPERATION

David Mayweg¹, Mattias Thuvander¹, Johan Eriksson¹

¹*Chalmers University of Technology, Sweden*

Zirconium alloys are used as fuel cladding in water-cooled and -moderated nuclear power reactors. The main degradation mechanisms are irradiation damage by fast neutrons, corrosion and hydrogen pick-up (HPU). We investigated a unique set of samples that were retrieved after 2082 days of operation in the commercial boiling water reactor *Oskarshamn 3*. Our analysis was aimed at improving the understanding of the degradation phenomena and we here present an overview of this work. The samples stem from cladding tubes that were manufactured from two alloys of slightly differing composition and we investigated two neutron fluences ($24 \cdot 10^{25} \text{ n m}^{-2}$ and $5.4 \cdot 10^{25} \text{ n m}^{-2}$). The alloys (*Zircaloy-2* and a slightly modified model alloy, 'Alloy 2') contain small amounts of Fe, Cr and Ni (0.18, 0.13, 0.06 wt% and 0.36, 0.18, 0.06 wt%, respectively) that improve corrosion properties and lower HPU. In addition to laser pulsing APT that is commonly employed on Zr and its alloys, we performed a significant number of voltage pulsing APT experiments, giving us improved crystallographic information [1], new insights regarding cluster shape [2] and better ability to detect Ni. We found a variety of clusters that are inhomogeneously distributed throughout the matrix and vary in size, shape and composition, and we show our attempts to characterize the clustering. Furthermore, we characterized the oxide/metal interface. Lastly, we try to shed light on hydrogen analysis, which is more limited than one would like – even employing cryo-FIB for specimen preparation.

[1] D. Mayweg, J. Eriksson, O. Bäcke, A.J. Breen, M. Thuvander, *Journal of Nuclear Materials* 581 (2023) 154444.

[2] J. Eriksson, G. Sundell, P. Tejlund, H.-O. Andrén, M. Thuvander, *Journal of Nuclear Materials* 550 (2021) 152923.

Nano-scale characterization of Nuclear and Radioactive Materials

5.4

ATOM PROBE TOMOGRAPHY TO INVESTIGATE IRRADIATION DAMAGE IN TUNGSTEN FOR NUCLEAR FUSION APPLICATIONS

Kieran C. Rivers¹, Andrew J. London², Dmitry Terentyev³, Michael P. Moody¹, Paul A.J. Bagot¹, David E.J. Armstrong¹

¹*University of Oxford, United Kingdom*

²*UK Atomic Energy Authority, United Kingdom*

³*Belgium Nuclear Research Centre, Belgium*

Tungsten is the main candidate for plasma facing components in future fusion reactor designs, but not enough is understood about how and why its macroscopic properties degrade when subjected to fusion neutron irradiation. Since no source of fusion neutrons exist for studying the microstructural changes in tungsten, this work involves using and analysing substitute irradiation sources. Specifically, high energy fission neutrons and self-ion irradiation are used in two separate studies which together will help explain tungsten's change in properties. Tungsten samples have been irradiated with fission neutrons in the BR2 SCK-CEN high-flux test reactor at 800°C to a dose of 0.8 dpa. In this study, Atom Probe Tomography (APT) is applied to characterise the microstructure of these samples at the atomic scale. Early findings show evidence of the formation of transmutant Re and Os atom clusters which will degrade mechanical properties of these irradiated materials. These findings will be compared to our existing work on samples neutron irradiated at higher temperatures to higher damage levels, allowing us to develop a better understanding of nucleation and growth processes in this material. Ion irradiation has benefits including allowing higher damage doses to be completed in shorter times to represent material degradation at later stages in fusion reactor lifetime. However, because ion irradiation cannot simulate the transmutation processes occurring in commercial fusion reactors, pre-alloyed ternary and quaternary compositions have been chosen for analysis, W-1.4Re-0.1Os and W-2.68Re-0.78Os-0.82Ta. APT analysis has not previously been carried out on a quaternary composition, which is crucial for understanding the long-term effects of fusion irradiation exposure with all the transmutation elements present. The samples were W self-ion irradiated to 1.7 dpa at 900°C. APT shows grain boundary decoration, clustering and solute decorated dislocation loops. The findings in neutron and ion irradiated samples will be compared and the usefulness and validity of using ion irradiation for understanding radiation induced microstructural evolution will be discussed.

Nano-scale characterization of Nuclear and Radioactive Materials

5.5

SYNERGETIC EFFECTS OF MN, NI, AND SI ON THE FORMATION OF MN-NI-SI CLUSTERS AND IRRADIATION HARDENING IN REACTOR PRESSURE VESSEL MODEL STEELS

Deepak Sharma¹, Auriane Etienne¹, Ronan Henry¹, Philippe Pareige¹, Bertrand Radiquet¹

¹*University of Rouen Normandie, France*

Tungsten is the main candidate for plasma facing components in future fusion reactor designs, but not enough is understood about how and why its macroscopic properties degrade when subjected to fusion neutron irradiation. Since no source of fusion neutrons exist for studying the microstructural changes in tungsten, this work involves using and analysing substitute irradiation sources. Specifically, high energy fission neutrons and self-ion irradiation are used in two separate studies which together will help explain tungsten's change in properties. Tungsten samples have been irradiated with fission neutrons in the BR2 SCK-CEN high-flux test reactor at 800°C to a dose of 0.8 dpa. In this study, Atom Probe Tomography (APT) is applied to characterise the microstructure of these samples at the atomic scale. Early findings show evidence of the formation of transmutant Re and Os atom clusters which will degrade mechanical properties of these irradiated materials. These findings will be compared to our existing work on samples neutron irradiated at higher temperatures to higher damage levels, allowing us to develop a better understanding of nucleation and growth processes in this material. Ion irradiation has benefits including allowing higher damage doses to be completed in shorter times to represent material degradation at later stages in fusion reactor lifetime. However, because ion irradiation cannot simulate the transmutation processes occurring in commercial fusion reactors, pre-alloyed ternary and quaternary compositions have been chosen for analysis, W-1.4Re-0.1Os and W-2.68Re-0.78Os-0.82Ta. APT analysis has not previously been carried out on a quaternary composition, which is crucial for understanding the long-term effects of fusion irradiation exposure with all the transmutation elements present. The samples were W self-ion irradiated to 1.7 dpa at 900°C. APT shows grain boundary decoration, clustering and solute decorated dislocation loops. The findings in neutron and ion irradiated samples will be compared and the usefulness and validity of using ion irradiation for understanding radiation induced microstructural evolution will be discussed.

Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software

6.1

DEVELOPMENT OF A REPOSITORY FOR APT GRAIN BOUNDARY EXCESS DATA

Alexander Reichmann¹, Christoph Dösinger¹, Daniel Scheiber², Vsevolod Razumovskiy², Markus Kühbach³, Lorenz Romaner¹

¹Montanuniversität Leoben, Austria

²Materials Center Leoben Forschung GmbH, Leoben, Austria

³Humboldt-Universität zu Berlin, Germany

Grain boundaries (GBs) play a key role in the design of high performance materials. A variety of experimental and theoretical methods exist to explore GB chemistry and structure. From the theoretical side, atomistic simulations provide a direct access to segregation energies, which are key fundamental GB properties and allow estimation of the Gibbs interfacial excess via thermodynamic models. On the experimental side, the most commonly applied technique to address GB excess nowadays is Atom Probe Tomography (APT). In order to consistently validate the predictions from theoretical simulations through the experimental data, consistent databases are required. While for Auger-electron spectroscopy a comprehensive database of GB segregation data has been compiled [1], a comparable database for APT data is currently missing. In this talk we will present our current activities regarding the creation of a database of APT GB excess data. In cooperation with the FAIRmat project we set up an online repository, which allows for sharing, publishing and analyzing APT GB data. The access and data management is organized through a NOMAD OASIS, which is provided with a python based tool-set to calculate excess given the reconstructed APT specimen. In addition, own data schemas have been developed for providing relevant metadata to the APT datasets, which are then stored using the community-developed NeXus file format (<https://fairmat-nfdi.github.io/nexus-fairmat-proposal>). The repository includes data from technologically relevant materials as, e.g. a Mo-Hf alloy [2] whose APT datasets can be directly investigated and analyzed. The community-driven approach is envisioned to continuously enlarge the database. Furthermore, in this talk, we also provide an overview of the computational methods available to predict the interfacial excess. Methods include in particular a combination of atomistic simulations and mean field methods, which can be closely validated with the experimental data. Furthermore, the potential for combining data-driven and physical models in a hybrid approach will be discussed.

[1] P. Lejček, Grain Boundary Segregation in Metals, Springer Berlin Heidelberg, Berlin, 2010

[2] K. Leitner, et al., How grain boundary chemistry controls the fracture mode of molybdenum, Materials and Design, 142, 36-43, 2018

Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software

6.2

EXTRACTING THERMODYNAMICS INFORMATION FROM LOCAL COMPOSITION FLUCTUATIONS IN SOLIDS: EXTENDED THEORY AND ITS APPLICATION TO SIMULATED AND EXPERIMENTAL ATOM PROBE DATA

Jianshu Zheng¹, Marvin Poul², Vsevolod Razumovskiy², Daniel Bitter¹, Patrick Stender¹, Guido Schmitz¹, Sebastian Eich¹

¹*University of Stuttgart, Germany*

²*Max-Planck-Institute für Eisenforschung GmbH, Germany*

While thermodynamic fluctuation theory has been applied to liquids for decades to obtain direct thermodynamic information, i.e. Gibbs free energies, from local composition fluctuations, the present work extends this theory to solids by considering an additional elastic work term which does not arise in liquids. This extended theory is firstly verified via atomistic simulations in an exemplary Cu–Ni embedded-atom system using Monte Carlo simulations at a fixed temperature over the entire composition range. Composition fluctuations in the system, which are tracked in various-sized subvolumes over time, reveal a systematic dependence on the size of the evaluation volume due to surface artefacts. Nonetheless, these surface artefacts can be excluded by extrapolation to an infinitely large subvolume, leading to perfect agreement with the prediction by the extended theory. Thus, recovering Gibbs free energy of mixing is possible. Atom Probe Tomography (APT) can determine local composition fluctuations by spatial frequency distribution analysis from high-resolution chemical and three-dimensional spatial information. The applicability of our approach is tested using simulated and experimental datasets of the Cu–Ni system. This methodology promises to improve the accuracy of thermodynamic information (e.g. miscibility gap, mixing/demixing tendencies, critical solution temperature) from direct atom probe measurements.

Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software

6.3

TOWARDS LARGE-SCALE FIELD EVAPORATION SIMULATIONS WITH AB INITIO ACCURACY

Shyam Katnagallu¹, Baptiste Gault¹, Patrick Stender¹, Jörg Neugebauer¹, Christoph Freysoldt¹

¹Max-Planck-Institute für Eisenforschung GmbH, Germany

In atom probe tomography (APT), electrostatic simulations at the atomic scale have proven to be crucial in understanding and evaluating reconstructions. However, purely electrostatic simulations do not capture interatomic interactions that occur on the surface. Recent attempts have combined molecular dynamics (MD) simulations with electrostatics [1] to better reproduce experimentally observed features. In these simulations, empirical interatomic potentials, which are fitted from electric-field free data, are augmented with an independent electrostatic force model. A conceptual alternative is density functional theory (DFT), which naturally combines interatomic and electrostatic long-range interactions and their dependence on high electric fields. Our results show that the potential energy surface experienced by an evaporating atom can become extremely shallow as a function of the electric field, leading to different possible evaporation pathways. While ab initio accuracy is necessary for accurate simulations, the computational cost of these calculations makes them prohibitive for large-scale simulations. To address this issue, we propose the use of machine learning interatomic potentials with an adaptive electrostatic component that redistributes the surface charge as atoms rearrange, and train these potentials from DFT data. We validate the Rappe et al [2] charge equilibration model using DFT reference charges acquired via Hirshfeld decomposition from metal slabs under electric fields ranging from 1 to 5 V/Å. We then develop interatomic potentials for field evaporation to demonstrate the performance and accuracy of our approach. These potentials can provide a better understanding of the complex interplay between electrostatic and interatomic interactions, leading to a more accurate representation of the evaporation process. Further research is needed to optimize these methods and apply them to larger systems, but our preliminary results are promising and highlight the potential of these approaches for advancing the understanding of field evaporation processes in APT.

[1] J. Qi, C. Oberdorfer, W. Windl, and E. A. Marquis, Ab Initio Simulation of Field Evaporation, *Phys. Rev. Mater.* 6, (2022).

[2] A. K. Rappe and W. A. G. Iii, Charge Equilibration for Molecular Dynamics Simulations, *J. Phys. Chem* 95, 3358 (1991).

Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software

6.4

COMPENSATING IMAGING DISTORTIONS IN A COMMERCIAL REFLECTRON TYPE ATOM PROBE

Martina Heller¹, Benedict Ott¹, Peter Felfer¹

¹Friedrich-Alexander Universität Erlangen-Nürnberg, Germany

In atom probe tomography, the spatial resolution and accuracy of the data critically depends on the 3D reconstruction of the 2D detector data. In atom probes with mass resolving power increasing reflectron mirrors, this must include a model of the imaging properties of the reflectron. However, for modern wide-angle reflectron instruments, these imaging properties are not trivial and need to be determined for the reflectron used. This is typically done by the instrument manufacturer and due to the proprietary nature of the instrument design is opaque to the user. In this paper, we provide a method to determine the imaging properties of a reflectron that can easily be carried out on commercial instrumentation. This method is used to provide the user with a transformation function from raw detector data to a virtual detector placed before the reflectron. From there on, 3D reconstructions can be carried out analogous to straight flight path instruments. Reference data for CAMECA LEAP 3000, 4000, 5000 and 6000 Series is also provided.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

7.1

ATOM PROBE TOMOGRAPHY FOR PREDICTING TIMELINES OF BIOCERAMIC BONE SCAFFOLD INTEGRATION IN VIVO

Natalie Holmes¹, Thomas Quenan², Iman Roohani¹, Julie M. Cairney¹, Hala Zreiqat¹

¹*University of Sydney, Australia*

²*Ecole Normale Supérieure Paris, France*

We recently reported the first atom probe study of the atomic-scale composition of in vivo bone formed in a bioceramic scaffold (strontium-hardystonite-gahnite)¹ after 12-month implantation in a large bone defect in sheep tibia.² This study detected elements from the degrading bioceramic implant, particularly aluminium (Al), in both the newly formed bone and in the original mature cortical bone tissue at the perimeter of the bioceramic implant. The study combined atom probe and nanoSIMS to enable a full picture of the bone tissue growth in the presence of the bioceramic to be constructed. As the next step, atom probe tomography is now uniquely placed to quantify the biodegradation rate of bioceramic scaffolds in vivo. Here we report our endeavours in this space, by beginning with the comparison of pristine bioceramic materials to 12-month in vivo degraded bioceramic implants. Quantifying the degradation rate, and associated kinetics, of such bone implants will allow us to predict the timepoint in which the bioimplant will fully degrade and the cavity will be fully replaced by natural bone tissue. The goal is to establish atom probe as a new tool for predicting timelines of synthetic bone scaffold integration in vivo, a new asset for the biomedical device industry.

[1] J.J. Li, et al. *Adv. Healthc. Mater.* 2019, 8, 1801298. [2] N.P. Holmes, et al. *Acta Biomater.* 2023, In Press.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

7.2

ADVANCEMENTS IN 3D NANOSCALE CHARACTERIZATION OF ORGANIC-MINERAL INTERFACES IN DENTAL ENAMEL

Jack Grimm¹, Sandra Taylor¹, Cameron Renteria², Bojana Ginovska¹, Dwayne Arola², Arun Devaraj¹

¹*Pacific Northwest National Laboratory, USA*

²*University of Washington, USA*

Despite being composed of a relatively hard, brittle mineral (hydroxyapatite, HA; $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), dental enamel survives decades of rigorous use in a challenging oral environment without cellular repair. The fundamental unit of enamel is calcium-deficient, carbonate-substituted HA nanocrystals, approximately 50 nm wide, 100 nm thick, and several millimeters in length. Each nanocrystal is separated by a thin (several nanometers) amorphous mineral phase, rich in organics. Atom probe tomography (APT) is suitable for this scale of characterization. Here, we present the value of atom probe tomography to study 1) the early stages of enamel formation 2) the growth mechanisms of defected enamel as well as 3) the aging mechanisms of enamel. During enamel formation (amelogenesis), organic matrix and amorphous mineral phases are secreted simultaneously. The interaction of those phases evolves as various proteins are cleaved and removed while the amorphous mineral phase transforms to HA and coarsens. We successfully conducted a systematic APT analysis of amelogenin protein coated on an HA microcrystal surface and at interrupted stages of amelogenesis.[1] Defected enamel can form due to a variety of genetic defects. Mouse models have shown that the absence of matrix metalloproteinase-20 (MMP20), for example, yields a thin and weak enamel with dramatically increased organic content surrounding loosely embedded HA nanocrystals. We used APT to analyze the nanoscale structure of these MMP20 mouse enamel. The outermost enamel surface becomes markedly harder and more prone to fracture as one ages. This is caused by ion exchange associated with de-/re-mineralization cycles altering the composition of the crystalline and amorphous intergranular phases. Using APT, we resolved the increase of mineral substituents (e.g. F and CO₃) and their spatial distribution (intergranular and core of the HA crystal, respectively) associated with increased age, revealing critical insights into the atomic scale mechanism of enamel aging. Additional efforts have been made to improve the confidence in the findings presented above. These include comparisons of the APT-measured composition of alternative calcium phosphate phases and the approximation of the field evaporation of HA and enamel.

[1] Taylor, S., et al. *Mat Adv Today* 18 (2023, in press)

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

7.3

TOWARDS NEAR-NATIVE STATE 3D ANALYSIS OF REHYDRATED E. COLI BACTERIAL CELLS USING ATOM PROBE TOMOGRAPHY

Florant Exeriter¹, Mohammed Al-Saady², Jing Fu², Ross K.W. Marceau¹

¹*Deakin University, Australia*

²*Monash University, Australia*

Understanding the structure and composition of microscopic organisms at the molecular to atomic scale is fundamental to solve crucial modern health crises (e.g., antibiotic resistance, the COVID-19 pandemic, etc). Atom probe tomography is a three-dimensional characterisation technique that is well-placed to offer combined structural and compositional analysis at the near-atomic scale, however there are significant challenges regarding samples from biological systems. In this study, pure water is graphene-encapsulated on top of a needle-shaped sample containing a section of dried Escherichia coli to (1) partially rehydrate the section of the bacterial cell and (2) enable voltage-pulsed atom probe tomography analysis in the frozen hydrated state. This novel sample preparation method enables a pathway for the collection of structural and chemical data from biological samples and opens a pathway towards near-atomic scale analysis of near-native state hydrated biological materials.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

7.4

SITE-SPECIFIC PREPARATION AND ATOM PROBE TOMOGRAPHY OF BULK WATER ICE AND AMINO ACID SOLUTIONS

Eric V. Woods¹, Leonardo Shoji Aota¹, Shuo Zhang¹, Tim M. Scharz¹, Mahander Pratap Singh¹, James O. Douglas², Baptiste Gault¹

¹Max Planck Institute für Eisenforschung, Germany

²Imperial College, UK

Overall, analyzing the structure of bio-molecules at near-atomic scale resolution while providing specific chemical information for each atom is critical to understanding their interactions with other molecules, metal cations, etc.. Atom probe tomography (APT) potentially could provide structural detail available via cryo-electron microscopy with spatially resolved elemental composition, including e.g. metal cations that are challenging to otherwise detect.

In this presentation, we will first discuss how to prepare aqueous solutions using GIS-free focused ion beam (FIB) lift-out protocol. We used our cryogenic sample workflow to first plunge freeze aqueous solutions on a supporting metal substrate, transfer them to a cryogenic gallium FIB system, prepare sharpened needle-like ice specimens on standard APT microtip arrays using site-specific lift-out techniques with redeposition welding. The final, sharpened samples are then transferred to the APT system for analysis, all under ultra-high vacuum conditions.

The resultant data demonstrate that controlling the electrical field magnitude is critical, because low electric field produce large water clusters $(H_2O)_nH^+$ with n up to X which can overlap with signals from organic species and therefore reduce the resolution and detectability. Optimal run conditions can largely suppress higher water clusters, allowing clearer identification and study of the fragmentation pattern of individual amino acids. Fragmentation and ionization of single, different amino acid solutions are presented to illustrate how critical selecting the proper parameters are for analyzing aqueous solutions, and how this will provide improved understanding for reconstructing actual protein structure via atom probe tomography.

[1] Woods, Eric *et al.* "A versatile and reproducible cryo-sample preparation methodology for atom probe studies." arXiv:2303.18048 [physics.app-ph]

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

7.5

CRYO ATOM PROBE TOMOGRAPHY STUDIES OF 5CB, 8CB LIQUID CRYSTALS

Kuan Meng¹, Patrick Stender¹, Guido Schmitz¹

¹*University of Stuttgart, Germany*

The development of Cryo-FIB enabled studies of various delicate samples of high interest, such as SEI [1], ferritin [2], porous borosilicate [3] or bio-molecules in aqueous solution [4]. Generally, organic materials exhibit complex mass spectra and a strong dependence on measurement conditions. Following our earlier measurements of frozen liquids, we are studying the evaporation and fragmentation of different liquid crystals. In detail, 5CB (4-Cyano-4'-pentylbiphenyl) and 8CB (4-Cyano-4'-octylbiphenyl) were chosen for investigation due to their relatively simple chemical structure and hydrophobically-induced stability. The liquid crystal tip preparation was improved based on the previous water and n-alkane study. Firstly, the liquid was dipped onto a pre-made tungsten cryo post [5] before freezing. Adjacent, the “multi-window” shaping technique [6] was used to shrink the frozen bulk into a proper size before adopting annular milling, in order to obtain wrinkle- and protrusion-free frozen liquid tips with a measurable hemisphere. Both types of molecules, pure 5CB and 8CB, evaporate predominantly as intact molecules, which suits our previous observation in the n-tetradecane study [5]. As a consequence, these two types of molecules can be distinguished even in the mixture of both elements, due to their pronounced mass difference. The dependence of mass spectra on the evaporation conditions is discussed and the miscibility of both species is explored in 3D reconstructions, which should enable the measurement of liquid mixtures. References:

- [1] Se-Ho Kim et al. (2022). Understanding the Degradation of a Model Si Anode in a Li-Ion Battery at the Atomic Scale. *The Journal of Physical Chemistry Letters*, 13(36), 8416–8421.
- [2] Qiu, S. et al. (2020). Graphene encapsulation enabled high-throughput atom probe tomography of liquid specimens. *Ultramicroscopy*, 216, 113036.
- [3] Perea, D.E. et al. (2020). Tomographic mapping of the nanoscale water-filled pore structure in corroded borosilicate glass. *npj Mater Degrad*, 4, 8.
- [4] Schwarz, T.M. et al. (2021). 3D Sub-Nanometer Analysis of Glucose in an Aqueous Solution by Cryo-Atom Probe Tomography, *Scientific reports* 11, 11607.
- [5] Schwarz, T.M. et al. Field evaporation and atom probe tomography of pure water tips. *Sci Rep* 10, 20271 (2020).
- [6] Meng, K. et al. (2022). Frozen n-Tetradecane Investigated by Cryo-Atom Probe Tomography. *Microscopy and Microanalysis*, 28(4), 1289-1299.

Nano-scale characterization of Geological and Planetary Materials

8.1

EARTHQUAKES AND ATOMS: NANOSCALE INSIGHTS INTO FAULT EVOLUTION

Emily M. Peterman¹, Steven M. Reddy², Alexis K. Ault³, David Saxey², Denis Fougerouse², Jordan L. Jensen³

¹*Bowdoin College, USA*

²*Curtin University, Australia*

³*Utah State University, USA*

High strain rate deformation in Earth's crust results in the formation of faults along which earthquakes rupture. During an earthquake, transient friction-generated heat (e.g., temperatures >1000 °C) can control deformation mechanisms and thus fault strength. Thin (0.1–1 mm-thick) mirror-like surfaces (fault mirrors) comprising nanoscale, polygonal hematite (Fe₂O₃) and/or partially amorphous silica suggest transient temperature rise during high strain rate deformation. Fault mirrors are ideal materials to investigate the nanoscale textural and geochemical response of rocks to earthquake processes and how fault strength evolves throughout the earthquake cycle. Here we use atom probe tomography (APT) to characterize geochemical and structural variations in two hematite fault mirrors from the Wasatch fault zone (Utah, USA). APT datasets of 25M to 100M detected ions inform the evolution of these materials during earthquakes. APT specimens prepared orthogonal to the fault mirror reveal several compositionally distinct features. In all specimens, Ca and Ti segregate to the grain boundaries of polygonal hematite crystals. These elements also decorate intragrain features interpreted as isolated dislocations. Some hematite crystals contain high concentrations of Ti and low Al concentrations. High concentrations of Si (+Al, Ca) are commonly located at triple junctions between hematite grains. Lastly, c. 30 nm clusters of a separate phase consisting of Si + Fe (+K, Ca) occur in a regular, repeating sequence within hematite. These nanoscale features are consistent with high-temperature nucleation and rapid crystal growth by grain boundary migration to produce polygonal hematite. Expulsion of trace elements as part of this process yielded Ca + Ti segregations along grain boundaries. Although some Si + Fe (+Al, Ca) remains trapped within clusters, most Si was expelled to triple junctions, which are energetically favorable for elements relatively incompatible in hematite. The paucity of decorated dislocations within the APT data contrasts with new transmission electron microscopy observations that show a plurality of dislocations. The relative absence of trace element decorated dislocations suggests limited diffusive transport after grain growth and indicates most dislocations post-date crystallization, thereby providing constraints on the timing of different earthquake processes. These findings may yield insight into high strain rate deformation processes in other materials and ceramics.

Nano-scale characterization of Geological and Planetary Materials

8.2

A NANOSCALE VISION OF PRIMITIVE METEORITES: APT CHARACTERIZATION OF THE FINE-GRAINED MESOSTASIS

Rick Verberne¹, Jean Bollard¹, Steven Reddy², David Wallis³, David Saxey², Martin Bizzarro¹

¹*University of Copenhagen, Denmark*

²*Curtin University, Australia*

³*University of Cambridge, UK*

Primitive undifferentiated meteorites consist of chondrules and are the precursor material for planetary objects. Chondrules are routinely studied to infer information about their age and processes taking place during the early stages of our solar system. Radiogenic Pb dating of Northwest Africa 5697 has constrained the timing of their formation to the first two million years after the formation of the solar protoplanetary disk. During this period, chondrules underwent reworking under high temperatures followed by low-temperature metamorphism during accretion of the parent body. Therefore, chondrules can inform about conditions and processes taking place during formation of our solar system. However, chemical and Pb-isotope analyses of chondrules are often performed with techniques that have low spatial resolution and therefore cannot deconvolve contributions from different components of the challenging, fine-grained structures. Chondrules in NWA 5697 have been dated using a bulk step-leaching process coupled with thermal ionization mass spectrometry. This approach limits our ability to retrieve information that is preserved within the fine-grained textures. Radiogenic Pb was shown to be present within the mesostasis; a fine-grained microcrystalline material with amorphous matrix and this raises the question whether initial U was present within submicrometre minerals or dispersed throughout the amorphous matrix. Furthermore, the radioactive decay of U and low-temperature metamorphism can lead to a redistribution of Pb and trace elements. Information not retrievable with the currently applied methods. Here, we focus on the nanoscale characterization of NWA 5697 chondrules. Electron backscatter diffraction combined with energy-dispersive X-ray mapping is used to characterize the structure and composition of submicrometre minerals within the mesostasis. Transmission electron microscopy and transmission Kikuchi diffraction were applied to identify structural nanoscale features and identify nanoscale phases within the mesostasis. These techniques are complemented by atom probe tomography to provide insight into the chemical and isotope composition and distribution at the nanoscale. The results show a heterogeneous distribution of Al and Si in the matrix and the presence of metallic inclusion. Importantly, we found no evidence for the redistribution of elements within the mesostasis. The lack of evidence for any trace-element mobility of crystallization demonstrates the robustness of Pb-Pb dating of mesostasis in chondrules.

Nano-scale characterization of Geological and Planetary Materials

8.3

NANOSCALE CONSTRAINTS ON THE EARLY-EARTH GEODYNAMO

Steven M. Reddy¹, Richard Taylor², David Saxey¹, Richard J. Harrison³, Will D. A. Rickard¹, Fengzai Tang⁴, Cauê S. Borlina⁵, Roger R. Fu⁶, Benjamin P. Weiss⁷, Paul Bagot⁸, Helen M. Williams³

¹*Curtin University, Australia*

²*Carl Zeiss Microscopy, Germany*

³*University of Cambridge, UK*

⁴*Warwick University, UK*

⁵*John Hopkins University, USA*

⁶*Harvard University, USA*

⁷*Massachusetts Institute of Technology, USA*

⁸*University of Oxford, UK*

The time of initiation of Earth's geodynamo has fundamental implications for planetary evolution and the ability of Earth to sustain life. Magnetite inclusions within detrital zircon grains older than 4 billion years are a potential record of the early Earth's geomagnetic field that may constrain geodynamo development. However, correctly interpreting the magnetic records from zircon grains is contentious due to the difficulty in quantifying the timing of magnetite formation. Here we utilize APT to investigate Fe mobility within highly magnetic zones of a 4-billion-year-old zircon from Jack Hills. Discrimination of subtle Fe signatures in mineral silicates can be difficult with APT due to interferences at 28 Da. However, analysis of $^{28}\text{Si}^{++}/^{28}\text{Si}^{+}$ and $^{54}\text{Fe}/^{56}\text{Fe}$ ratios provide a means of identifying a true Fe signal within the APT reconstructions. The APT data from the magnetic zones of the zircon sample show the presence of Pb-bearing nanoclusters (~10 nm in diameter) that record elemental compositional variations consistent with formation during two discrete thermal events at 3.4 billion years ago and < 2 billion years ago. The older population of clusters contain no detectable Fe. In contrast, Fe-bearing clusters all belong to the younger cluster population. The results indicate that the Fe required to form secondary magnetite was not present in the zircon prior to 3.4 Ga and that remobilization of Pb and Fe, the latter associated with magnetite formation, took place after 2 Ga, more than one billion years after deposition of the Jack Hills sediment at 3 Ga. The results demonstrate the novel capability of APT to yield direct age constraints on Fe mobility in ancient minerals and provide a framework for future testing of the age of the palaeomagnetic signal recorded in early-Earth zircon.

Nano-scale characterization of Geological and Planetary Materials

8.4

CONTAINMENT OF RADIONUCLIDE CONTAMINANTS IN MINERAL INTERFACES

David Saxey¹

¹Curtin University, Australia

The effective capture and removal of fugitive radionuclides represents a major challenge to remediation of mine sites, waste sites, or nuclear accidents. The ability of layered double hydroxide, anionic clay minerals known as hydrotalcites (HTC) to sequester a range of contaminants from solution offers a unique potential remedy. However, HTC do not provide a robust repository for actinide isolation over the long term. In this study[1], we formed HTC by in-situ precipitation in a barren lixiviant (aqueous solution) from a uranium mine and thermally transformed the resulting radionuclide-laden product. After thermal decomposition of the nanoscale HTC, atom probe tomography and other analytical methods were employed to characterize the mineralogical changes. Nanoscale mineral phases were observed highly concentrated in U, Th and REE as analogues of other actinides and daughter radionuclides. The resulting minerals, such as a U-Th-REE mineral, and a V-rich apatite mineral, have the potential to host radionuclides and actinides. The periclase, spinel-, and olivine-group minerals, which constitute >99.9% of the final mineralogy, have highly favourable geochemical and P-T geotechnical properties and inherent resistance to radiation damage, making them suitable for disposal in a nuclear waste repository. U-rich phases associated with the mineral interfaces record a U concentration factor of ~ 50,000 relative to the original solute demonstrating high extraction and concentration efficiencies. Importantly, no radionuclides were detectable in the periclase, spinel- and olivine-group minerals, thus allowing the opportunity for specific recovery and reprocessing of the inter-granular, radionuclide-enriched becquerelite and V-rich apatite phases. These results record the efficient sequestering of radionuclides from contaminated water and this novel, broad-spectrum, nanoscale HTC capture and concentration process constitutes a rapid solute decontamination pathway and solids containment option in perpetuity.

[1] Douglas, G. B., Reddy, S. M., Saxey, D. W., MacRae, C. M., Webster, N. A. S., & Beeching, L. J. (2023). Engineered mineralogical interfaces as radionuclide repositories. *Scientific Reports*, 13(1), 2121.

Nano-scale characterization of Metals and Alloys

9.1

ATOM PROBE TOMOGRAPHY AND IN-SITU SYNCHROTRON STUDY OF A PRECIPITATION HARDENED STEEL

Mattias Thuvander¹, Magnus Hörnqvist Colliander¹, Severin Jakob¹, Steve Ooi²

¹*Chalmers University of Technology, Sweden*

²*Ovako Group R&D, Sweden*

The relatively new steel called Hybrid Steel (from Ovako AB) is a high-performance martensitic steel with high strength, good corrosion properties, and good resistance towards softening when used at elevated temperatures. The main composition is 5Cr-6Ni-2Al-0.5V-0.7Mo (wt%) with 0.18 wt% C. The strength relies on the concomitant formation of nanometer-sized intermetallic B2-NiAl, Cr-rich carbides, and V-rich carbides. In this study, we have employed in-situ synchrotron diffraction and small angle X-ray scattering (SAXS) characterization during heat treatment up to 7 h. This was complemented by atom probe tomography (APT) after certain heat treatments. The synchrotron results show how the size and volume fraction of the B2-NiAl evolve during heat treatment. The in-situ diffraction data also shows that the amount of austenite is higher during heat treatment than after cool-down. The APT measurements show that the carbides are always in contact with B2 particles, but not vice versa. In addition to Ni and Al, the B2 particles are also enriched in Mn and Cu. It is also shown that the carbides coarsen faster than the B2 particles, so probably the B2 particles are responsible for the good resistance towards softening.

Nano-scale characterization of Metals and Alloys

9.2

MAPPING THE INFLUENCE OF DENDRITIC SEGREGATION ON THE NANO-SCALE IN SINGLE CRYSTAL NI-BASED SUPERALLOYS

Victoria C. I. Strutt¹, Michael P. Moody¹, Paul A. J. Bagot¹, Matthew Appleton², Jane Woolrich²

¹*University of Oxford, United Kingdom*

²*Rolls-Royce plc, United Kingdom*

Nickel-based superalloys are widely used for the fabrication of high temperature turbine blades, owing to their remarkable capabilities at high temperature and under stress[1]. The two-phase microstructure, consisting of a gamma (γ) matrix and gamma prime (γ') precipitates, gives these alloys their excellent mechanical properties and is achieved through selective alloying. Numerous studies have shown that many such alloying elements demonstrate a preference for either the γ or γ' phase to varying extents, e.g. Al and Ta into the γ' phases and Cr, Re and Co into the γ matrix[2][3]. Whilst alloying additions are necessary to achieve the desired microstructure, the differences in the physical and chemical properties of these elements also results in solute partitioning between dendrite cores (DCs) and interdendritic regions (IDRs). Despite extensive heat treatment, studies have shown residual segregation between DCs and IDS persists[4][5], with additional studies linking variation in mechanical properties between both dendrite cores and interdendritic regions to changes in local chemistry[6][7]. This study compares the variation between DCs and IDRs in TMS138a, a 4th generation superalloy. The high resolution of atom-probe tomography allows for the quantification of differences in local chemistry of γ and γ' phases, nano-scale precipitates and interfacial behaviour between DCs and IDRs. The local chemistry in ex-service alloys is further studied on the nano-scale and correlated to variations between DCs and IDRs. Understanding and characterising the effects of residual microsegregation in these alloys will optimize future alloy design and ensure the stability of these materials under increasingly demanding conditions.

[1] R. Darolia, *Int. Mater. Rev.*, 64 (2019) 355, doi:10.1080/09506608.2018.1516713.

[2] A. Heckl et al., *Mater. Sci. Eng. A*, 528 (2011) 3435, doi:10.1016/j.msea.2011.01.023.

[3] P. J. Bocchini et al., *Scr. Mater.*, 68 (2013) 563, doi:10.1016/j.scriptamat.2012.11.035.

[4] A. Szczotok et al., *J. Mater. Eng. Perform*, 23 (2014) 2739, doi:10.1007/s11665-013-0843-1.

[5] M. Karunaratne et al., *Superalloys 2000 (Ninth International Symposium)*, (2000) 263, doi:10.7449/2000/superalloys_2000_263_272.

[6] R. C. Reed et al., *Mater. Sci. Technol.*, 23 (2007), doi:10.1179/174328407X192723.

[7] G. X. Lu et al., *Philos. Mag. Lett.*, 96, (2016) 461, doi:10.1080/09500839.2016.1252860.

Nano-scale characterization of Metals and Alloys

9.3

EXPLORING AVENUES FOR TAILORING THE THERMAL STABILITY OF HIERARCHICAL MICROSTRUCTURES IN NI-BASED SUPERALLOYS VIA PHASE TARGETED ALLOY DESIGN

Florian Vogel¹, Mingshuai Huang², Michael Pavel³, Mark Weaver³, Yuan Wu⁴, Weiwei Zheng⁴, Sieglind Ngai¹, Yuxiang Lai¹, Pan Xie¹, Jianghua Chen¹

¹Hainan University, China

²Jinan University, China

³University of Alabama, USA

⁴University of Science and Technology Beijing, China

The performance of Ni-based high-temperature structural materials for critical components in jet engines and gas turbines, is defined by the alloys' high-temperature properties and microstructural stability. Performance, service life and sustainability of such alloys are critically intertwined and controlled by microstructure and phase chemistry. Ni-based superalloys comprise a γ matrix (A1) with cube-shaped γ' precipitates (L1₂), a hierarchical microstructure is created when additional nanoscale γ particles emerge within γ' precipitates. Such hierarchical microstructures show a prominent impact on mechanical and high-temperature creep properties of Ni-based superalloys [1,2]. However, research has identified two metastability pathways: The γ particles either emerge as spheres and then transform to plates which further grow and split γ' precipitates, or they grow and then gradually dissolve within γ' precipitates. Both scenarios result in a loss of the strengthening effect. Such behavior is determined by phase chemistry and intrinsically linked to thermodynamics, i.e. enthalpy of mixing of γ' , elastic energy and interface energy, and kinetics controlled by diffusivity. Here, we explore the impact of adding γ forming elements to Ni_{86.1-n}Al_{8.5}Ti_{5.4}X_n with X = Cr, Co, Mo, Ru, W, Hf or Re and n = 1–4 at.%, on formation and thermal phase stability of hierarchical microstructures. We show that phase targeted alloy design by adding γ forming species enables to specifically trigger partitioning to the hierarchical γ particles, and thereby to control their morphology and thermal stability. We utilized transmission electron microscopy (TEM), atom probe tomography (APT) and ThermoCalc to ascertain the fundamental mechanisms underpinning the formation and thermal stability of hierarchical microstructures in the context of thermodynamics and kinetics. We compare our experimental phase chemistry data to predictions made by ThermoCalc. Our work demonstrates avenues for enhancing the thermal stability of hierarchical microstructures via phase targeted alloy design.

[1] F. Vogel *et al.*, Mapping the evolution of hierarchical microstructures in a Ni-based superalloy, *Nature Communications* (2013) 4:2955, (DOI: 10.1038/ncomms3955)

[2] Y. Chen *et al.*, Hierarchical microstructure strengthening in a single crystal high entropy superalloy, *Scientific Reports* (2020) 10:12163, (DOI: 10.1038/s41598-020-69257-8)

Nano-scale characterization of Metals and Alloys

9.4

A THOROUGH STUDY OF GRAIN BOUNDARY SEGREGATION, ENERGY, AND WIDTH IN COPPER–NICKEL THIN FILMS USING ATOM PROBE TOMOGRAPHY

Rüya Duran¹, Felix Fischer¹, Sebastian Eich¹, Patrick Stender¹, Guido Schmitz¹

¹University of Stuttgart, Germany

In this work, grain boundary (GB) segregation in the copper–nickel system is analyzed via atom probe tomography (APT) for various bulk compositions at a temperature of 700 K. GB segregation is characterized by means of APT measurements in correlation with FIB/tEBS (Focused Ion Beam/transmission Electron Backscatter Diffraction) techniques. Four exemplary alloy compositions with nickel contents of 25 at.%, 60 at.%, 70 at.%, and 85 at.% were deposited onto tungsten posts through ion beam sputtering and subsequently heat treated at 700 K for 24 h to reach thermodynamic equilibrium. Segregation of copper to the GBs was observed for all bulk compositions and quantified for a total of about 70 individual GBs in terms of the solute excess Γ . These excess values are used within an analytic model of the Gibbs isotherm in order to determine the change in GB formation energy through segregation over the entire composition range. Eventually, the GB segregation widths were determined from the individual composition profiles across the GB. To compensate for density fluctuations observed in the reconstructed atom probe data, a short molecular dynamics simulation was performed to equilibrate the atomic distances, resulting in a uniform density profile across the GB. Considering a representative GB within the nanocrystalline GB network based on the complete dataset of about 70 GBs, the width is consistently evaluated to be approximately 4.5 nm, although the segregation value varies, i.e. no significant dependence of the GB width on the solute excess Γ was found for the investigated bulk compositions. A similar qualitative trend over a wide range of intermediate compositions could be confirmed in accompanying atomistic simulations within the embedded-atom framework for two representative $\Sigma 5$ and $\Sigma 3$ symmetrical tilt GBs.

Nano-scale characterization of Metals and Alloys

9.5

NANOSCALE CHARACTERISATION OF POWDER PROCESSED NI ODS ALLOYS

Megan Carter¹, Michael Moody¹, Peter Hosemann², Djamel Kaoumi³, David Armstrong¹

¹*University of Oxford, UK*

²*University of California, USA*

³*University of North Carolina*

An ongoing collaboration between the University of Oxford, University of North Carolina and UC Berkeley is investigating the development of oxide dispersion strengthened (ODS) Ni-based alloys for potential use in Molten Salt Reactors (MSR). New Ni alloys are needed to provide the combination high-temperature strength, tolerance to irradiation, and corrosion resistance required by certain components in next generation MSRs. To this end, ODS Ni-based alloys are a possible solution. The incorporation of oxide particles has been proposed as a method for trapping He, which is a transmutation product of irradiated Ni and is known to cause grain boundary embrittlement. Furthermore, the oxide particles also improve creep resistance. This work focuses on manufacturing Ni-based ODS alloys using powder metallurgy. Hastelloy N powder is used as the base material and combined with oxide-forming additions (Yttria and Zr/Ti). APT was used to analyse the extent to which different starting powder compositions, milling parameters and consolidation methods influence the nature of ODS inclusions within the Ni alloys. In particular, the impact of cryomilling is presented. Elemental analysis of cryomilled powders was conducted with APT and SEM-EDX. Specifically, we highlight the role of cryomilling in introducing contaminant elements into mechanically alloyed powders and the knock-on effect this has on the microstructure and ODS inclusions in the consolidated alloy. Furthermore, results from additive manufacture (3D printing) as a consolidation method for these materials is compared to those obtained via the more conventional sintering route, with respect to ODS number density, morphology and composition. Characterisation results from APT and STEM-EDX are compared, and, combined with nanohardness results, provide feedback on which processing routes show the most promise for ODS-Ni alloy development.

Nano-scale characterization of Metals and Alloys

9.6

MORPHOLOGICAL AND CHEMICAL EVOLUTION OF NANOSCALE PRECIPITATES IN ALLOY 718 AS A FUNCTION OF PROCESSING

Vitor Vieira Rielli¹, Sophie Primig¹

¹*University of Sydney, Australia*

Advanced understanding of the evolution of nanoscale precipitates in Ni-based superalloys during processing and in service has often been enabled through the use of atom probe microscopy (APM). These complex materials find widespread applications in high temperature settings, such as in jet engines. Improving their performance is essential for developing more fuel-efficient next generation aircrafts. The critical role of the perhaps most commonly used aerospace grade Alloy 718 is due to its high mechanical strength, which is mainly attributed to the presence of nanoscale γ' and γ'' precipitates. Certain desirable complex morphologies are engineered via thermo-mechanical processing or, more recently, additive manufacturing followed by thermal treatments. For example, unique configurations of γ' and γ'' precipitates can be generated during the so-called direct ageing route, leading to a 10% increase in yield strength when compared to conventional solution annealing and ageing [1]. This talk will give an overview on the complex correlation between macro- and microstructural components of Alloy 718 introduced during various processing routes including cast-and-wrought and additively manufactured parts [2,3]. We focus on the morphology and composition of strengthening precipitates analyzed via APM, discuss their evolution during conventional and direct ageing, and elucidate links between hot forging and heterogeneities in an industrially forged turbine disk [2,4]. Furthermore, we show that the dislocation density generated during hot compression directly affects the stacking sequence of co-precipitates [5]. Finally, we present a non-lift-out sample preparation procedure that allows straightforward micro-manufacturing of site-specific atom probe tips, with considerably lower likelihood of premature tip fracture.

[1] F. Theska, A. Stanojevic, B. Oberwinkler, S.P. Ringer, S. Primig, *Acta Mater.* 156 (2018) 116–124.

[2] V.V. Rielli, F. Godor, C. Gruber, A. Stanojevic, B. Oberwinkler, S. Primig, *Mater. Des.* 212 (2021) 110295.

[3] V.V. Rielli, A. Piglione, M.S. Pham, S. Primig, *Addit. Manuf.* 50 (2022) 102540. [4] V.V. Rielli, F. Theska, F. Godor, A. Stanojevic, B. Oberwinkler, S. Primig, *Mater. Des.* 205 (2021) 109762.

[5] V. V Rielli, F. Godor, C. Gruber, A. Stanojevic, B. Oberwinkler, S. Primig, *Scr. Mater.* 226 (2023) 115266.

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

10.1

ANALYSIS OF ICE-LOADED NANOPOROUS COPPER TIPS USING ATOM PROBE TOMOGRAPHY

Levi Teqqi¹, Ingrid E. McCarroll², Eric Woods², Renelle Dubosq², Se-Ho Kim², Baptiste Gault², Julie M. Cairney¹

¹*University of Sydney, Australia*

²*Max-Planck-Institut für Eisenforschung GmbH, Germany*

Atom probe tomography provides three-dimensional microanalysis at near-atomic resolution, but extending the technique from metals and semiconductors to soft matter and liquids faces challenges related to sample preparation and data interpretation [1]. Here we report on the use of nanoporous copper to support the analysis of water ice in the atom probe. A Cu-Mn alloy is dealloyed using hydrochloric acid [2], rinsed with distilled water, plunge-frozen in liquid nitrogen, then cryo-transferred to a cryo plasma focused ion-beam for milling, then cryo-transferred to the atom probe for analysis. This presentation will mainly cover interpretation and analysis of the reconstructed datasets. Regions of copper and ice are clearly observed, respectively evaporating mainly as Cu^+ and $\text{H}(\text{H}_2\text{O})^+$. Mass resolution of the ice peaks is significantly improved over similar studies [3] due to the enhanced electric field delivered by the continuous copper framework. Larger water clusters such as $\text{H}(\text{H}_2\text{O})_2$ are more abundant in the middle of the pores, reflecting the reduced field intensity far from the copper ligament. Concentration profiles over the metal-ice interface show increase in copper oxide and hydroxide peaks, suggesting this method could be used for analysis of metal-liquid interfaces, as well studies into frozen liquids and solutions.

[1] I. E. McCarroll et al. *Materials Today Advances* 7, 100090 (2020).

[2] L.-Y. Chen et al. *Advanced Functional Materials* 19, 1221–1226 (2009).

[3] P. Stender et al. *Microscopy and Microanalysis* 28, 1150–1167 (2022).

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

10.2

CRYO-ATOM PROBE TOMOGRAPHY TO INVESTIGATE FROZEN LIQUIDS IN TEMPLATED MATERIALS

Oliver Waszkiewicz¹, Eric V. Woods², Baptiste Gault^{1,2}, Ifan E. L. Stephens¹, Mary P. Ryan¹

¹Imperial College London, UK

²Max-Planck-Institut für Eisenforschung GmbH, Germany

The electric double layer (EDL) describes the interfacial region between solid and electrolyte liquid phases where the charge distribution is non-homogenous [1]. This region is fundamentally significant to all electrochemistry, as it is in these interfacial regions where electrochemical reactions occur. However, these interfaces are highly complex, dynamic systems with length scales in the 10s of nanometres and despite advances in modelling simulations in recent years, discrepancies between theory and experimental results remain [2]. Recently developed cryogenic-atom probe tomography (cryo-APT) methods have demonstrated the capability of freezing liquid-solid interfaces and subsequently analysing them in the atom probe providing near atomic-scale atom maps at the solid-liquid interface [3,4]. However, so far, no studies have attempted to push cryo-APT further towards the analysis of the EDL. Major obstacles include the difficulty of obtaining high freezing rates necessary to preserve the EDL within vitrified ice. To develop systematic analyses of EDL's for studying electrochemical reactions, we aim to implement new advancements in freezing protocols and sample preparation for the cryo-APT investigations of sodium iodide solutions frozen within nanoporous anodised aluminium oxide (AAO) substrates. The use of high-pressure freezing for cryo-APT will be explored for the first time; combined with prior electrodeposition of Cu monolayers on AAO to investigate the effect of pore-surface decoration on the arrangement of sodium and iodide ions.

[1] Zbigniew Stojek. "The Electrical Double Layer and Its Structure". In: *Electroanalytical Methods: Guide to Experiments and Applications*. Ed. by Fritz Scholz. Berlin, Heidelberg: Springer, 2002, pp. 3–8. isbn: 978-3-662-04757-6. doi: 10.1007/978-3-662-04757-6_1.

[2] Doblhoff-Dier and Marc T. M. Koper. "Electric double layer of Pt(111): Known unknowns and unknown knowns". In: *Current Opinion in Electrochemistry* 39 (June 1, 2023), 101258. issn: 2451-9103. doi: 10.1016/j.coelec.2023.101258.

[3] A. El-Zoka et al. "Enabling near-atomic-scale analysis of frozen water". In: *Science Advances* 6.49 (Dec. 4, 2020). Publisher: American Association for the Advancement of Science, eabd6324. doi: 10.1126/sciadv.abd6324.

[4] Patrick Stender et al. "Status and Direction of Atom Probe Analysis of Frozen Liquids". In: *Microscopy and Microanalysis* 28.4 (Aug. 2022). Publisher: Cambridge University Press, 1150–1167. issn: 1431-9276, 1435-8115. doi: 10.1017/S1431927621013994.

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

10.3

DEEP UV-LASER ASSISTED ATOM PROBE TOMOGRAPHY ANALYSIS OF GLASS MATERIALS FOR PROTEIN ENCAPSULATION

Gustav Eriksson¹

¹*Chalmers University of Technology, Sweden*

The three-dimensional structure of proteins is an important piece of information when studying biological systems. While the amino-acid sequence of proteins easily can be identified, the folding of proteins cannot be determined from this alone. Computational methods based on machine learning to predict protein structures have improved in the last years but is still relying on experimental to validate the results. Several endeavours to develop methodology to characterize proteins with APT have been undertaken in the last years. One such method is based on the encapsulation of proteins in a silica matrix formed by a sol-gel process¹. This method allows for the retention of the protein's native structure and conformation in their hydrated state in a material solid at room temperature, enabling manipulation of the material and sample preparation without the need for cryogenic techniques. However, one major limitation of silica analysis using APT is the yield of the analysed specimen. The combination of the material's insulating properties, mechanical fragility under the field induced stresses and poor absorption of the laser makes the specimen prone to premature fracture during analysis. By using the new Cameca LEAP 6000 XR equipped with a deep UV-laser ($\lambda = 257.5$ nm), silica has been analysed yielding a larger number of collected ions before specimen fracture than what is obtained with a longer wavelength laser. A systematic study of the experimental parameters shows the effect of laser energy on the charge state ratio, mass resolving power, compositional accuracy and type of molecular ion species that are evaporated. This information gives insight into the evaporation behaviour of silica under the effect of a deep-UV laser and can be used to better understand these materials. The results show that the use of a deep-UV laser opens up the possibility to study materials previously difficult to study due to poor absorption of the laser at longer wavelengths. Successful analysis of these type of materials can open up avenues to make protein characterization by APT more readily available.

Sundell, G., Hulander, M., Pihl, A. & Andersson, M. Atom Probe Tomography for 3D Structural and Chemical Analysis of Individual Proteins. *Small* **15**, 1–6 (2019).

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

10.4

CORRELATIVE IN-SITU LIQUID CELL ELECTROCHEMISTRY TEM AND CRYOGENIC APT OF LIQUID-SOLID INTERFACES

Shelly Conroy¹, James Douglas¹, Neil Mulcahy¹, Mary Ryan¹, Baptiste Gault^{1,2},

¹Imperial College London, UK

²Max-Planck-Institut für Eisenforschung GmbH, Germany

Liquid cell transmission electron microscopy(LCTEM) allows for solution phase dynamic processes to be visualised and probed at the nanoscale at unprecedented temporal and spatial resolutions in comparison to other liquid microscopy techniques such as light and physical probe microscopes. Additionally MEMS based biasing sample holder chip design has provided a platform to perform electrochemical experiments to replicate battery cycling experiments within the electron microscope. While these experiments have been useful for gaining a micro and nanoscale in-situ understanding of electrochemical processes that occur at solid-liquid interfaces of battery materials, the current spatial resolution of liquid processes within the TEM is still not capable of resolving and quantifying complex dynamic nanoscale structures at atomic resolutions. As a result, the performance of electrochemical systems is currently limited due to a lack of understanding of complex interactions that occur between mobile species and electrode materials. Finding a correlative high-resolution characterisation method which is compatible with both the liquid and solid component of the interface is of great importance to gain a fundamental understanding of these processes at an atomic level. Cryogenic focused ion beam(FIB) preparation and atom probe tomography(APT) has allowed researchers to investigate the native vitrified liquid-solid interfaces of samples at the near to atomic scale. In this presentation we will present how one can combine in-situ electrochemistry LCTEM with site specific cryogenic atom probe tomography. We investigate Li based solid electrode interfaces and dendrites formed during the electrochemical cycling. While there have been numerous examples in recent years of room temperature correlative TEM and APT, there has been no such examples of correlative LCTEM-APT or correlative cryogenic TEM-APT. The primary reason for this is the difficulty in transferring, preparing, and maintaining specimens in cryogenic conditions. In this study the liquid in the LCTEM chips are cryogenically vitrified following in-situ electrochemical LCTEM experiments, in order to allow APT needle samples to be created from the liquid-solid interface of interest. Recent advances in vacuum cryogenic transfer module technology, and cryogenic FIB site specific sample preparation workflows, have allowed for this type of correlative cryogenic multi-microscopy approach to be realised.

Technical Advances in Specimen Preparation and (cryo) Transfer, Instrumentation, Workflow Automation

10.5

A REACTOR CHAMBER ATTACHED TO THE ATOM PROBE TOMOGRAPHY FOR ENABLING IN SITU STUDIES OF SOLUTE PARTITIONING TO INTERFACES DURING HEAT TREATMENTS AND OXIDATION

Arun Devaraj¹, Sten Lambeets¹, Dallin Barton¹, Elizabeth Kautz¹, Bharat Gwalani¹, Amit Shyam², Jonathan Poplawsky², Daniel Perea¹

¹*Pacific Northwest National Laboratory, USA*

²*Oak Ridge National Laboratory, USA*

For designing alloys that are stable for high-temperature applications, it is critical to understand and control the solute partitioning that occurs at phase boundaries during heat treatments or high-temperature exposures. Similarly, when it comes to designing alloys for nuclear applications with a high propensity for corrosion, it is crucial to understand the elemental partitioning that occurs at the oxide-metal interfaces. At PNNL, a reactor chamber attached to a CAMECA LEAP 4000XR atom probe tomography (APT) system allows the study of solute partitioning during heat treatments and oxidation of alloys for temperatures up to 500 °C [1]. This talk will highlight the value of in-situ APT for monitoring solute partitioning in a promising high-temperature Al-Cu-Mn-Zr alloy, specifically to reveal atomic-scale mechanisms that lead to the emergence of non-equilibrium solute segregation to strengthening precipitate(θ')-matrix interface [2]. In addition, examples of the use of the in situ reactor chamber for studying elemental diffusion mechanisms across oxide-metal interfaces during oxidation of alloys including Zirconium alloys [3, 4], Uranium-molybdenum alloys [5], and model austenitic steels [6] will be presented.

[1] S. V. Lambeets et. al., Nanoscale perspectives of metal degradation via in situ atom probe tomography, *Topics in Catalysis*, 1-17, 2020

[2] B. Gwalani et. al, Rapid assessment of interfacial stabilization mechanisms of metastable precipitates to accelerate high-temperature Al-alloy development, *Materials Research Letters*, 10 (12), 771-779, 2022.

[3] Z Yu et. al., Irradiation Damage Reduces Alloy Corrosion Rate via Oxide Space Charge Compensation Effects, *Acta Materialia*, 253, 118956, 2023.

[4] E. J. Kautz et. al., Rapid assessment of structural and compositional changes during early stages of zirconium alloy oxidation, *NPJ Materials Degradation*, 4(1), 1-9, 2020

[5] E. J Kautz et. al., Compositional partitioning during early stages of oxidation of a uranium-molybdenum alloy, *Scripta Materialia*, 212, 114528, 2022

[6] A. Devaraj et. al., Visualizing the Nanoscale Oxygen and Cation Transport Mechanisms

during the Early Stages of Oxidation of Fe–Cr–Ni Alloy Using In Situ Atom Probe Tomography, Advanced Materials Interfaces, 2200134, 2022.

Nano-scale characterization of Metals and Alloys

11.1

RE-EVALUATING OUR UNDERSTANDING OF MAGNESIUM OXIDATION USING ATOM PROBE TOMOGRAPHY

Ingrid McCarroll¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

As we develop our scientific knowledge we lay out foundations for how we understand the world. As new scientific techniques are developed we obtain deeper insight into certain processes, occasionally revealing flaws in these scientific foundations. Over the past decade I have been applying the technique of atom probe tomography to the study of magnesium oxidation with rather surprising results. In this presentation I will detail the existing theories related to the early oxidation processes of magnesium alloys and outline discrepancies between these theories and results from atom probe studies of magnesium oxidation experiments.

Nano-scale characterization of Metals and Alloys

11.2

HIGH-TEMPERATURE OXIDATION OF ALLOYS: APT INSIGHTS INTO NANOSCALE MECHANISMS

Ivan Povstugar¹, Ashok Vayyala¹, Dmitry Naumenko², Andrey Litnovsky², Felix Klein², Timur Galiullin²

¹*Central Institute for Engineering, Jülich, Germany*

²*Institute of Energy and Climate Research, Jülich, Germany*

Oxidation and corrosion of engineering alloys cause substantial economic losses and can entail accident scenarios. For high-temperature applications, where the use of external protective coatings is limited, alloys with intrinsically high oxidation resistance are needed. Usually, such alloys protect themselves under service conditions via formation of a surface oxide scale, typically Cr_2O_3 or Al_2O_3 , which limits further oxidation. Protective properties (growth, adhesion, permeability etc.) of oxide scales are greatly dependent on the alloy composition, including minor constituents, as well as on the oxidizing atmosphere. Understanding the oxidation mechanisms is critical for further alloy development and requires knowledge of the oxide scale microstructure and chemistry down to the nanoscale level. Here we will overview the application of APT to extensive systematic characterization of chromia and alumina scales grown on several alloy classes intended for high-temperature applications, such as ferritic FeCr(Al) steels, Co-based Haynes alloys and W-based SMART alloys. Thorough analysis and accurate quantification of the local elemental compositions by APT, especially for interfacial and grain boundary segregations of minor alloy constituents (Ti, Mn, Nb, Ta etc.) and the so-called reactive elements (La, Y), is shown to be decisive for understanding the mechanisms of oxide scale growth in complex engineering alloys. Quantitative comparison of the local chemistry of oxide scales in a series of FeCr steels revealed the role of individual minor constituents in the acceleration of mass transport through the oxide, and eventually explained deteriorated oxidation resistance. The use of ^{18}O isotope tracer in an oxidizing atmosphere coupled with unique APT capabilities directly visualizes pathways for oxygen diffusion, unveils hidden details of microstructural evolution of the oxide scale, which are essential for the correct interpretation of experimental data obtained from conventional “larger-scale” analytical techniques, and proves itself as a powerful tool for studies of oxidation mechanisms.

Nano-scale characterization of Metals and Alloys

11.3

HIGH-TEMPERATURE OXIDATION AT Nb- Al_2O_3 INTERFACES AT THE NANOSCALE

Michael Eusterholz¹, Torben Boll¹

¹*Karlsruhe Institute of Technology, Germany*

The electrification of high-temperature processes, such as steel casting, can remarkably increase energy efficiency and reduce costs. Lining parts like slide plates and spider bricks must withstand temperatures above 1500°C during service and are commonly made from conventional refractory ceramics, e.g., Al_2O_3 . While they excel in corrosive and oxidative environments, they frequently suffer from failure caused by thermomechanical stresses. By incorporating an electrically conductive component, composites of refractory metals and ceramics enable resistive preheating of the parts, significantly reducing the thermal gradients. Al_2O_3 with either Nb or Ta are appropriate candidates due to their thermal expansion behavior. Such composites are commonly fabricated by pressureless sintering, which takes place at temperatures in the range of those during service. While a protective atmosphere is used to exclude O-containing gases, previous research showed that different refractory metal oxides form in the microstructure of sintered specimens [1]. To specifically study the formation of such phases, we prepare model materials by sputter-coating of $\alpha\text{-Al}_2\text{O}_3$ with Nb, which are then heat-treated at 1600°C with minimal O uptake (Ar, graphite-lined furnace, 2h, 10 Kmin^{-1}). Our complementary approach of transmission electron microscopy (TEM) methods and atom probe tomography (APT) reveals how Nb oxide forms along Nb grain boundaries and at triple junctions with the Al_2O_3 -Nb interface, a ternary oxide evolves [2]. Furthermore, we modify the Nb film to adjust the oxidation at the Al_2O_3 -Nb interface and Nb grain boundaries.

Nano-scale characterization of Metals and Alloys

11.4

REVISING NANOSTRUCTURE AND MAGNETISM OF $\text{SM}(\text{CO,ZR})_{7.5}$ MAGNETS

Nikita Polin¹, Konstantin Skokov², Esmail Adabifiroozjaei², Alex Aubert², Hongguo Zhang³, Burçak Ekitli², Oliver Gutfleisch², Leopoldo Molino-Luna², Dierk Raabe¹, Baptiste Gault^{1,4}

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

²Technische Universität Darmstadt, Germany

³Beijing University of Technology, China

⁴Imperial College London, UK

Permanent magnets are key components for mechanical-electric energy conversion in devices like hard disc drives, electric cars and wind turbines [1]. Besides NdFeB magnets, the $\text{Sm}(\text{CoFeCuZr})_{7\pm\delta}$ magnets are the main high energy density permanent magnets on the market whereas the latter outperform for high temperature applications and aggressive chemical environments. The performance of such magnets strongly depends on the interplay of composition and distribution of their constituting phases and structural defects on different length scales. However, the quaternary $\text{Sm}(\text{CoFeCuZr})_{7\pm\delta}$ magnet is highly complex in terms of the microstructure and composition on the nanoscale which makes it difficult to establish a straight forward structure-property relationship. Here we investigate binary $\text{SmCo}_{7.5}$ and ternary $\text{Sm}(\text{Co,Zr})_{7.5}$ magnetic alloys across different length scales by electron microscopy and atom probe tomography. By reducing number of alloys' constituents, we can resolve the contribution of each constituent to the magnetic performance.

[1] O. Gutfleisch, M. A. Willard, E. Brück, C. H. Chen, S. G. Sankar, and J. P. Liu, "Magnetic materials and devices for the 21st century: Stronger, lighter, and more energy efficient," *Adv. Mater.*, vol. 23, no. 7, pp. 821–842, 2011.

Nano-scale characterization of Metals and Alloys

11.5

CARBON CONTROLLED GROWTH OF COLUMNAR FERRITE NANOSTRUCTURES IN IRON CARBON THIN FILMS

Jonas Arlt¹, Moritz Roscher¹, Tobias Meyer¹, Thomas Brede¹, Christine Borchers¹, Tim M. Schwarz², Reiner Kirchheim¹, Cynthia A. Volkert¹

¹University of Göttingen, Germany

²University of Stuttgart, Germany

The iron carbon system shows complex non-equilibrium microstructures, for example during the decomposition of cementite under severe plastic deformation [1] or during film deposition at low temperatures [2]. In the present work we combined four-dimensional scanning transmission electron microscopy (4D-STEM) [3] with the chemical resolving capability of atom probe tomography (APT) to gain deep insights into the microstructural evolution and carbon distribution of sputtered iron carbon thin films. Iron carbon films of varying carbon concentration were prepared by argon plasma sputtering at 100°C. Prior to 4D-STEM and APT analysis films were pre-characterized by X-ray diffraction, electron probe microanalysis and conventional TEM. While films generally consist of columnar ferrite for low and are largely amorphous for high carbon concentrations, we observe a complex evolution in microstructure from substrate to film surface for intermediate concentrations. In a first layer of amorphous iron carbon, ferrite nuclei form and act as seeds for further columnar growth of ferrite throughout the rest of film to the surface. Columns become thinner as they approach the surface. Moreover, our APT results show that neighboring columns are separated by carbon rich regions. The intermediate concentration case shows a remarkable example of film self-structurization on the nanoscale and allows for a retrospective study of the growth kinetics in terms of distance from substrate. We propose a growth model to explain the different stages of film evolution based on the decisive role of carbon. Furthermore, we discuss the complex interplay between carbon and defect structure in context of the defactant concept [4] as well as in comparison with other carbon induced and stabilized structure transformations like cementite decomposition in cold wire drawing [1] or abnormal grain growth under electromigration of carbon [5].

[1] Y. J. Li et al., *Acta Materialia* **84** (2015)

[2] A. Weck et al., *J Mater Sci* **47** (2012)

[3] C. Ophus, *Microscopy and Microanalysis* **25** (2019)

[4] R. Kirchheim, *International Journal of Materials Research* **100** (2009)

[5] T. Brede et al., *Advanced Engineering Materials* **n/a** (2023)

Nano-scale characterization of Metals and Alloys

11.6

PROBING DEALLOYING MECHANISMS IN THIN-FILM NANOPOROUS METALS USING ATOM PROBE TOMOGRAPHY

Ezgi Hatipoğlu¹, Aparna Saksena¹, Jochen M. Schneider², Baptiste Gault^{1,3}, Ayman A. El-Zoka^{1,3}

¹*Max-Planck-Institut für Eisenforschung GmbH, Germany*

²*RWTH Aachen University, Germany*

³*Imperial College London, UK*

Dealloying corrosion has become a popular method for obtaining highly functional nanoporous gold (NPG) substrates[1]. The selective dissolution of Ag from AgAu solid solutions in acidic environments leads to the formation of an open-pore, bicontinuous 3D, nanoporous structure. Using thin film precursors, as opposed to solid-solution alloys, facilitates easier integration of NPG into energy storage and catalytic devices owing to the inherent substrate-support feature[2] [3]. High-resolution studies of dealloyed thin films could guide optimization efforts by revealing the effect of as-deposited structure and dealloying conditions on the morphology and chemical structure of dealloyed substrates. In this investigation, we focus on studying NPG made by dealloying AgAu thin films deposited via magnetron sputtering. Using scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD), we uncovered the different morphologies attained due to non-equilibrium AgAu compositions created by the sputtering process. Atom probe tomography (APT) was used to study the influence of dealloying conditions on nanoligament compositions, at the atomic-scale[4]. We correlate our findings to subtleties in the dealloying mechanism of AgAu, that are introduced by the thin-film properties of AgAu. Significant findings include the different electrochemical regimes of dealloying and the formation of Au-rich segregates at grain boundaries.

[1] Erlebacher, J., Newman, C. & Sieradzki, K. Fundamental physics and chemistry of nanoporosity evolution during dealloying. *RSC Nanosci. Nanotechnol.* 11–29 (2012). doi:10.1039/9781849735285-00011

[2] Henkelmann, G. et al. Self-Detachment and Subsurface Densification of Dealloyed Nanoporous Thin Films. *Nano Lett.* 22, 6787–6793 (2022).

[3] Plawsky, J. L., Kim, J. K. & Schubert, E. F. Engineered nanoporous and nanostructured films. *Mater. Today* 12, 36–45 (2009).

[4] El-Zoka, A. A., Langelier, B., Botton, G. A. & Newman, R. C. Enhanced analysis of nanoporous gold by atom probe tomography. *Mater. Charact.* 128, (2017).

Nano-scale characterization of Semiconductor Materials and Devices

12.1

PHOTONIC ATOM PROBE STUDY OF SUB-BANDGAP LUMINESCENCE IN A GAN/INGAN HETEROSTRUCTURE

Lorenzo Rigutti¹, Eric Weikum¹, Abraham Diaz Damian¹, Jonathan Houard¹, Gérald da Costa¹, Fabien Delaroche¹, Angela Vella¹, Grzegorz Muziol², Henryk Turski²

¹University of Rouen Normandie, France

²UNIPRESS, Warsaw, Poland

The Photonic Atom Probe (PAP) allows for the measurement of Photoluminescence (PL) of a sample tip while it is being analyzed by Laser-Assisted Atom Probe. The femtosecond Laser pulse required for the La-APT measurement also serves to excite the free charge carriers, whose recombination provide the PL signal. As a consequence, it becomes possible to correlate the optical signature of the different parts of a complex structure with the 3D distribution of the contained chemical species [1]. We present an application of the PAP on III-nitride p-i-n junctions, namely a thick (> 1 μ m thickness) novel multi-layer structure containing InGaN quantum wells and a buried tunnel junction [2]. Plasma-assisted molecular beam epitaxy-grown structures, which were used in the study, are characterized by diverse doping concentrations in both p- and n-type regions. The PL spectra can be correlated with the 3D chemical information from APT [3]. The PL signals exhibit indeed a donor-acceptor pair (DAP) emission, whose spectral features can be related to the 3D distribution of the Mg dopants and provide information about the stress induced by the applied electric field during the experiment [4]. In particular, it becomes possible to correlate the DAP peak energy with the Mg concentration in different layers. Furthermore, the APT analysis shows that the Mg distribution is random. These results open interesting perspectives for studies of light-emitting defects at the nanoscale.

[1] J. Houard et al., Review of Scientific Instruments 91.8 (2020): 083704.

[2] H. Turski, et al., ECS J. Solid State Sci. Technol., 9 (2020) 015018.

[3] E. Di Russo et al., Nano Lett. 2020, 20, 12, 8733–8738.

[4] P. Dalapati et al., Physical Review Applied 15.2 (2021): 024014.

Nano-scale characterization of Semiconductor Materials and Devices

12.2

EXPLORING STACKING FAULTS IN GAN-BASED LEDS THROUGH APT INVESTIGATION

Ruiying Shu¹, Rachel A. Oliver², Martin Frentrup², Menno J. Kappers², David J. Wallis^{2,3}, Christina Hofer¹, Paul A. J. Bagot¹, Michael P. Moody¹

¹University of Oxford, UK

²University of Cambridge, UK

³University of Cardiff, Wales

GaN-based light-emitting diodes (LEDs) are widely used in the lighting industry due to their high external quantum efficiency. Current efficient LED bulbs are based on blue-emitting structures as green emitters show much lower efficiency ^[1]. Zincblende InGaN-based emitters have been predicted to offer more efficient green emission because of a smaller bandgap and a lack of polarization fields ^[2], however, intrinsic {111} stacking faults are commonly found in these structures. These extended planar defects may lead to local variations in indium content in the quantum wells, potentially reducing the spectral purity and influencing device efficiency ^[3]. Therefore, characterizing the structure and composition of these stacking faults is essential for understanding how the microstructure of zincblende GaN can influence LED performance. As a powerful tool for studying materials at the atomic scale, atom probe tomography (APT) offers unparalleled spatial resolution and chemical sensitivity. This study used APT to investigate the atomic-scale compositional variation within quantum wells and the commonly occurring stacking faults in zincblende nitrides. Specifically, we optimized the operational parameters of APT with a particular focus on their effect on quantitative measurements. We then investigated the chemical composition of the stacking faults and their surrounding regions to gain insights into the mechanisms of their formation and their impact on the performance of emitters. The findings of this study provide valuable information on the influence of stacking faults in zincblende nitrides.

[1] C. J. Humphreys, J. T. Griffiths, F. Tang, F. Oehler, S. D. Findlay, C. Zheng, J. Etheridge, T. L. Martin, P. A. J. Bagot, M. P. Moody, D. Sutherland, P. Dawson, S. Schulz, S. Zhang, W. Y. Fu, T. Zhu, M. J. Kappers and R. A. Oliver, *Ultramicroscopy* 2017, 176, 93-98.

[2] D. R. Elsaesser, M. T. Durniak, A. S. Bross and C. Wetzel, *Journal of Applied Physics* 2017, 122, 115703.

[3] B. Ding, M. Frentrup, S. M. Fairclough, M. J. Kappers, M. Jain, A. Kovács, D. J. Wallis and R. A. Oliver, *Journal of Applied Physics* 2020, 128, 145703.

Nano-scale characterization of Semiconductor Materials and Devices

12.3

EXTREME ULTRAVIOLET PULSED ATOM PROBE TOMOGRAPHY OF β -Ga₂O₃

Luis Miaja-Avila¹, Benjamin W. Caplins¹, Jacob M. Garcia¹, Ann N. Chiaramonti¹, Rebecca L. Peterson², Norman A. Sanford¹

¹*National Institute of Standards and Technology, USA*

²*University of Michigan, USA*

Ga₂O₃ is an attractive ultra-wide bandgap semiconductor material for applications in optoelectronics and high-power devices [1]. In this work, we perform compositional APT studies on unintentionally doped β -Ga₂O₃ using a commercial laser-pulsed APT instrument and a custom-built extreme ultraviolet (EUV) APT. While the commercial near-UV (NUV) APT system utilizes a 355-nm laser to induce field ion evaporation, our custom system replaces the near-ultraviolet laser in a straight flight-path atom probe microscope with a coherent EUV light source and a vacuum beamline. With these modifications, we are able to illuminate the specimen's tip with 30-45 eV photons [2]. In our EUV APT measurements we maintain a constant ion detection rate while varying the EUV pulse energy by approximately two orders of magnitude (x120) and sample standing voltage. Our results show an underestimation of the oxygen composition when performing APT experiments under high EUV pulse energy or low field conditions. It is determined that compositional values closer to the expected nominal stoichiometry are obtained when performing experiments under high field conditions. Our results are similar to recently published results with a NUV APT instrument [3]. These published NUV APT experiments were performed at 100 kHz laser repetition rate, for this reason we performed additional studies with a commercial NUV APT instrument at 25 kHz. A careful compositional analysis comparison between the NUV and EUV systems will be discussed. Additionally, we identify possible dissociation-related peaks in our NUV and EUV mass spectra. These peaks have been previously overlooked or misidentified in the literature. Finally, our compositional analysis shows Si concentration values orders of magnitude higher than the values obtained through secondary-ion mass spectrometry. This disconnect is of importance as technologically-relevant Ga₂O₃ devices with Si implantation are under development [4].

[1] M. Higashiwaki et al., *Semicond. Sci. Technol.* 31, 034001 (2016)

[2] A.N. Chiaramonti, L. Miaja-Avila, P.T. Blanchard, et al., *MRS Advances* 4, 44-45 2367 (2019)

[3] F. Chabanais, E. Di Russo, A. Karg, et al., *Microscopy and Microanalysis* 27, 687 (2021)

[4] M. Higashiwaki, *Phys. Status Solidi RRL* 15, 2100357 (2021)

Nano-scale characterization of Semiconductor Materials and Devices

12.4

A FIELD DEPENDENT STUDY ON THE COMPOSITIONAL ANALYSIS OF BORON DOPED SILICON USING A GRID AND NORMAL LOCAL ELECTRODE.

Richard J. H. Morris¹, Ramya Cuduvally², Jeroen E. Scheerder¹, Mattias Thuvander³, Claudia Fleischmann¹⁴

¹IMEC, Belgium

²McMaster University, USA

³Chalmers University, Sweden

⁴KU Leuven, Belgium

Boron has been the p-type dopant of silicon-based semiconductors for decades. With device scaling, the conventional go-to analysis technique of secondary ion mass spectrometry (SIMS) is now approaching its application limit. However, atom probe tomography (APT) offers the potential to fill this role. As such, there have been many APT studies of boron doped Si but achieving reproducible and accurate results remains elusive [1, 2]. Recent work has shown that the APT analysis conditions, especially the apex field, can significantly impact upon the quantification [3, 4]. In this study we have therefore explored the impact of the apex electric field on boron doped Si quantification. A uniformly boron doped silicon sample benchmarked using SIMS and a NIST reference was investigated. Field dependent measurements were performed on our LEAP 5000 XR using a GRID- [4] and normal-electrode (NE) and by varying laser pulse energy within the range 30-85 pJ while retaining a constant evaporation rate. The GRID electrode is designed to remove co-evaporated ions, enabling their impact to be investigated. From the resulting data and for a Si charge state ratio (CSR) the boron was found to be underestimated. However, at this abruptly changes, and the boron now becomes overestimated. This change coincided with a transition from an inhomogeneous to homogeneous boron hit map. Increasing further i.e., up to 0.9, resulted in the boron decreasing, although it remained overestimated. This compositional behavior with CSR was found to be the same for both the NE and GRID electrodes. However, the variation in the boron composition determined as a function of Si CSR > 0.08 for the NE showed a more erratic behavior compared to the GRID electrode. The significantly reduced boron variation of the GRID analysis offered a more reproducible data set and enabled a clear trend with Si CSR i.e., apex field, to be observed.

[1] F Meisenkothen *et al*, *Ultramicroscopy* 159 (2015) 101-111.

[2] D. J. Larson *et al*, *Micosc. Microanal* 24 (suppl 1) (2018) 1084-1085.

[3] R. Cuduvally *et al*, *Ultramicroscopy* 210 (2020) 112918.

[4] R.J.H Morris *et al*, *Ultramicroscopy* 241 (2022) 113592.

Nano-scale characterization of Semiconductor Materials and Devices

12.5

QUANTITATIVE DOPANT ANALYSIS IN SILICON VIA ATOM PROBE TOMOGRAPHY

Karen DeRocher¹, Mark McLean¹, Frederick Meisenkothen¹

¹*National Institute of Standards and Technology, USA*

Atom probe tomography (APT) is currently the only characterization technique that may be capable of providing 3D dopant distributions in semiconductor devices at device dimensions. While APT can deliver chemical information down to concentrations of 10's of $\mu\text{g/g}$ (ppm_{at}), for some materials, electric field conditions in the atom probe can bias the quantitative analysis results [1, 2]. While calibration of data using reference materials is common in many characterization techniques, it is not often used in APT. However, we've found that a standards-based approach to APT analysis can dramatically improve the accuracy of composition measurements in phosphorous-doped silicon. Using a reference material with a known dose of phosphorous (NIST SRM 2133 [3]), we carried out APT measurements under a variety of analysis conditions and constructed a calibration curve we then applied to a sample doped with a known concentration of phosphorous. Comparing our composition measurement without correction to values obtained after application of the calibration curve showed an improvement from 26% relative error to less than 4%. We're now looking to extend this analysis approach to other industrially relevant dopants in Si, as well as additional materials systems in order to improve the accuracy and reliability of composition measurements made using APT [4].

[1] R. J. H. Morris, R. Cuduvally, D. Melkonyan, C. Fleischmann, M. Zhao, L. Arnoldi, P. van der Heide, W. Vandervorst, *Journal of Vacuum Science & Technology B* **36** (2018), p. 03F130-(1-6). doi: 10.1116/1.5019693

[2] DeRocher, K. , McLean, M. and Meisenkothen, F. (2022), *Microscopy and Microanalysis*, Portland, OR, US, [online], <https://doi.org/10.1017/S1431927622003373>

[3] D. S. Simmons, R. G. Downing, G. P. Lamaze, R. M. Lindstrom, R. R. Greenberg, R. L. Paul, *Journal of Vacuum Science and Technology B* **25** (2007), p. 1365-1375. doi: 10.1116/1.2759937

[4] Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

Advances in Hydrogen detection

13.1

A FULLY OPEN SOURCE TOOLCHAIN FOR ATOM PROBE TOMOGRAPHY OF HYDROGEN

Peter Felfer¹

¹Friedrich-Alexander Universität Erlangen-Nürnberg, Germany

Atom probe tomography has seen a vast increase in use over the past two decades, driven by innovations in the instrument hardware. These include drastically improved data collection rates, wider detection angles and the use of lasers enabled non-conducting specimens. Initially, these innovations were only possible by using high-end electronics and tight system integration. As a result, the majority of instruments were commercially made. With drastic increase in computational performance and speeds of widely available bus systems, these days even high-performance APT systems can be realized using off the shelf parts.

For groups that wish to conduct specialized APT experiments that demand custom instrumentation, significant barriers still are the lack of reference designs, the software needed to turn the available hardware into a working instrument and the software to analyze the acquired data. In this talk, we will introduce the open-source end-to-end toolchain developed at FAU Erlangen, that offers an open reference design, instrument control software and data analysis algorithms. The instrument reference design is that of a voltage pulsed micro-electrode setup with a straight flight path, that sits on top of an optical table. This leaves the option to add a laser, as is currently being done. The design is available upon request from the group, to make sure anyone wanting to build an instrument gets the design in the most suitable form for them. Along with the instrument design, we offer a Python based control software (<https://github.com/mmonajem/pyccapt>), that is able to control an APT experiment and record the data from two different detector vendors. Finally, we offer an open-source atom probe data analysis toolbox (<https://github.com/peterfelfer/Atom-Probe-Toolbox>) with a wide range of analysis algorithms implemented in scriptable form. This is complemented by an atom probe plugin for the open-source computer graphics suite Blender for high-end visualization and object modelling (<https://github.com/peterfelfer/AtomBlend-II>).

Advances in Hydrogen detection

13.2

HYDROGEN TRAPPING CAPABILITY OF HYBRID STEEL INVESTIGATED BY DEUTERIUM CHARGED SAMPLES IN ATOM PROBE EXPERIMENTS

Severin Jakob¹, Steve Ooi², Mattias Thuvander¹

¹*Chalmers University of Technology, Sweden*

²*Ovako, Sweden*

Atom probe tomography has seen a vast increase in use over the past two decades, driven by innovations in the instrument hardware. These include drastically improved data collection rates, wider detection angles and the use of lasers enabled non-conducting specimens. Initially, these innovations were only possible by using high-end electronics and tight system integration. As a result, the majority of instruments were commercially made. With drastic increase in computational performance and speeds of widely available bus systems, these days even high-performance APT systems can be realized using off the shelf parts.

For groups that wish to conduct specialized APT experiments that demand custom instrumentation, significant barriers still are the lack of reference designs, the software needed to turn the available hardware into a working instrument and the software to analyze the acquired data. In this talk, we will introduce the open-source end-to-end toolchain developed at FAU Erlangen, that offers an open reference design, instrument control software and data analysis algorithms. The instrument reference design is that of a voltage pulsed micro-electrode setup with a straight flight path, that sits on top of an optical table. This leaves the option to add a laser, as is currently being done. The design is available upon request from the group, to make sure anyone wanting to build an instrument gets the design in the most suitable form for them. Along with the instrument design, we offer a Python based control software (<https://github.com/mmonajem/pyccapt>), that is able to control an APT experiment and record the data from two different detector vendors. Finally, we offer an open-source atom probe data analysis toolbox (<https://github.com/peterfelfer/Atom-Probe-Toolbox>) with a wide range of analysis algorithms implemented in scriptable form. This is complemented by an atom probe plugin for the open-source computer graphics suite Blender for high-end visualization and object modelling (<https://github.com/peterfelfer/AtomBlend-II>).

Advances in Hydrogen detection

13.3

VISUALIZING RADIATION-INDUCED DAMAGE BY HYDROGEN/DEUTERIUM

Aparna Saksena¹, Binhan Sun¹, Xizhen Dong¹, Heena Khanchandani¹, Baptiste Gault¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

Atom probe tomography (APT) is now extensively used to measure the local chemistry of materials. Since the probed volume is usually quite small, with a starting apex diameter ≤ 50 nm, site-specific specimen preparation via a focused ion beam (FIB) is routinely implemented. This preparation route is often supplemented by transmission Kikuchi diffraction (TKD) to further position the region of interest, closer to the apex. Irradiating the specimen with the electron and ion beam could lead to the generation of vacancies and even amorphization of the specimen. These extrinsically created vacancies become crucial for probing H/D since, as it is well-known, vacancies act as a strong trap for H/D. Here, we investigated the feasibility of site-specific preparation of a medium Mn steel containing austenite (fcc) and ferrite (bcc). APT specimens were charged in a deuterium gas atmosphere, where the distribution of deuterium shows clusters of point defects after Pt deposition, conventional Ga-FIB preparation, and TKD which were enriched by up to 35 at.% deuterium. These deuterium-rich clusters are sequentially minimized by systematically eliminating these preparation-induced damages to unveil the intrinsic H/D traps in the material.

Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software detection

14.1

HIDDEN STRUCTURES IN ATOM PROBE TOMOGRAPHY DATA

Sebastian Koelling¹, Simone Assali¹, David N. Seidman², Dieter Isheim², Oussama Moutanabbir¹

¹*Polytechnique Montreal, Canada*

²*Northwestern University, Chicago, USA*

Atom Probe Tomography data are known to contain remnants of the atomic plane sets forming on the surface of the tip-shaped samples [1]. As APT analyses are scale-free, these plane sets are useful as they provide an internal scale that enables the calibration of the tomographic reconstruction of the acquired data [2]. Here, we will introduce an algorithm that allows us to reveal and track the presence of crystallographic remnants throughout entire atom probe data sets automatically. We will show that remnant planes are more abundant than previously known, can readily be used to implement dynamic reconstruction protocols on 5-10nm slices of APT data sets [3] and enable us to extract the dimension of features in the data set with an accuracy similar to Transmission Electron Microscopy.

[1] N.D. Wallace et al., Ultramicroscopy 189, 65 (2018).

[2] P. V. Liddicoat et al., Nature Communications 1, 1 (2010). [3] B. Gault et al., Ultramicroscopy 111, 1619 (2011).

Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software detection

14.2

AN INVESTIGATION INTO PROBABILISTICALLY ASSIGNING PEAK-OVERLAPPED IONS WHEN PERFORMING CLUSTER SEARCHES IN APT DATA

Benjamin Jenkins¹, Aidar Zakirov¹, Maria Vrellou¹, Philippe Pareige¹, Cristelle Pareige¹, Bertrand Radiguet¹

¹*University of Rouen Normandie, France*

Atom probe tomography (APT) is well-suited to study early-stage solute cluster formation in a variety of materials. To identify individual solute clusters in APT data, several approaches are widely implemented throughout the community, with the most commonly used being the maximum separation method and its variants[1,2]. All cluster search methods require the analyst to select a variety of parameters, which can lead to large user-to-user variation in results[3]. One key parameter required in each cluster search algorithm is which elements are included in the search step. This selection can be trivial, but mass-to-charge-state peak overlaps between matrix elements and those expected to be clustered complicates the situation (e.g. Ni clusters in Fe-Ni alloys). There is no consensus on how to process these peak overlaps and, despite proposed methods that rely on trying to determine the elemental identity of each ion[3], the most common approach remains to either include or exclude the entire overlapped peak in the cluster search, which is likely to bias experimental results. A possible approach is to randomly assign ions in the overlap peak between elements A and B as either element A or element B, based on the ratio of A:B in the overlap peak, similar to Ref.[4]. We expand upon previous work by performing the random assignment step many times to produce multiple datasets on which cluster searches can be performed. This enables the likelihood of each atom belonging to a cluster to be determined and permits a statistical approach to be taken. In this presentation, we will investigate the application of probabilistic assignment to multiple simulated datasets, with differing ratios of A:B in the peak overlap and varying cluster/matrix properties. Since the ground truth of each ion's mass-to-charge-state-ratio, elemental identity, and whether it is a cluster or matrix ion is known, we are able to determine the accuracy of our approach and will compare it to the traditional approaches of including/excluding the entire peak in both the maximum separation and iso-position cluster search methods. Our approach will also be applied to an experimental dataset, and its utility discussed.

[1] J.M.Hyde,C.A.English: Symposium R 'Microstructural Processes in Irradiated Materials', Fall_MRS,2000

[2] <https://doi.org/10.1017/s1431927607070900>

[3] <https://doi.org/10.1017/S1431927618015581>

[4] <https://doi.org/10.1017/S1431927616012782>

Advances in data reconstruction & data analysis, Machine learning Approaches, Simulation Tools, Open Software detection

14.3

MACHINE LEARNING-ENABLED TOMOGRAPHIC IMAGING OF CHEMICAL SHORT-RANGE ATOMIC ORDERING IN CoCrNi ALLOY

Yue Li¹, Baptiste Gault¹

¹Max-Planck Institut für Eisenforschung GmbH, Germany

In solids, chemical short-range order (CSRO) refers to the self-organisation of atoms of certain species occupying specific crystal sites. CSRO is increasingly being envisaged as a lever to tailor the mechanical and functional properties of materials. Yet quantitative relationships between properties and the morphology, number density, and atomic configurations of CSRO domains remain elusive. Herein, we showcase how machine learning-enhanced atom probe tomography (APT) can mine the near-atomically resolved APT data and jointly exploit the technique's high elemental sensitivity to provide a 3D quantitative analysis of CSRO in a CoCrNi medium-entropy alloy. We reveal multiple CSRO configurations, with their formation supported by state-of-the-art Monte-Carlo simulations. Quantitative analysis of these CSROs allows us to establish relationships between processing parameters and physical properties. The unambiguous characterization of CSRO will help refine strategies for designing advanced materials by manipulating atomic-scale architectures.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P1

ENHANCING YIELD, PERFORMANCE AND FIELD-OF-VIEW OF ATOM PROBE TOMOGRAPHY BY IN-SITU METALLIC COATING OF SPECIMENS

Tim M. Schwarz¹, Eric Woods¹, Mahander P. Singh¹, Chanwon Jung¹, Leonardo S. Aota¹, Kyuseon Jang¹, Se-Ho Kim^{1,2}, Ingrid McCarroll¹, Baptiste Gault^{1,3}

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

²Korea University, Republic of Korea

³Imperial College London, UK

Atom probe tomography requires sharp, needle-shaped specimens with a diameter typically below 100 nm, making them both very fragile and reactive, i.e. subject to facile degradation. Defects such as notches that can appear at grain boundaries or precipitates during specimen preparation, as well as reactions on the specimen's surface, are known to affect the yield and data quality. The use of a conformal coating directly on the sharpened specimen has been proposed to increase yield and improve background. However, to date, these coating have been applied ex-situ, which imposes transport of specimen through ambient atmosphere into a separate instrument, and the metallic film may not perfectly cover the entire surface. Here, we report on the controlled in-situ deposition of a thin metal film on specimens within the focused ion beam immediately after specimen preparation. The metallic targets of different metals (Cr, Ti and Ag) were attached to the micromanipulator via a conventional lift-out and sputtered by using the Ga or Xe focused ion beam. The ejected atoms and ions act as a source for depositing a thin, uniform and conformal thin film on the specimen. We showcase the many advantages of coating specimens from metallic and non-metallic materials, including an increase in data quality and yield, a reduction of the background and improved mass resolution, and an increase in the effective field-of-view. The ease of implementation of the approach makes it very attractive for generalizing its use across a very wide range of atom probe analyses.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P2

NUCLEARIZATION OF ATOM PROBE TOMOGRAPHY AND THE WORKFLOW ANALYSIS: ATOMIC-SCALE CHARACTERIZATION OF NEUTRON IRRADIATED MATERIALS

Coraline Hossepied¹

¹CEA Saclay; France

The LECI hotlab facility at CEA Saclay in France is equipped with a nuclearized APT in combination with APT specimen preparation for neutron-irradiated materials. The APT analysis needs a needle shape specimen (with curvature radius <50nm) by using either electropolishing or Focused Ion Beam (FIB). This information is especially important on neutron-irradiated material to characterize chemical composition variations around point defect clusters or grain boundaries. This work is a major stake for CEA/DRMP (Department of Materials and Physical Chemistry Research for Low Carbon Energy) that includes several steps, such as the manufacturing of a glove box, the instrumental nuclearization of the APT in association with the nuclear safety and radiation protection. This project includes also the nuclearization of sample preparation devices, integrated in shielded glovebox/cells, that is designed to prepare massive electropolished or FIB samples from radioactive material. The transfer and activity control between each unit from sample preparation devices to APT were also optimized in order to increase the final analyses yield. The presentation will highlight the impacting factors when designing a nuclearized APT, as well as the limitations and difficulties when undertaking such a project. The first analyses of radioactive material has been performed and several studies are already planned on different nuclear irradiated materials such as fuel cladding, reactor vessel... Some examples of such APT analysis will be presented.

This work has benefited from a state aid managed by the National Research Agency under the program "Investments for the future" with the reference ANR-11-EQPX-0020.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P3

CONTROLLED ENVIRONMENT WORKFLOWS FOR CRYO-APT

Urs Maier¹, David von Gunten¹, Claudio Weiss¹, Dave Piguet¹, Marc Maier¹, Tobias Vassalli¹, Max Hofmaenner¹, Cyril Straessle¹, Nicki Wehrli¹, Ljiridona Asani¹, Leo Scherrer¹, Stephan Michels¹, Janosch Buechi¹, Antonina Moroz¹, Tobias Hofmaenner¹, Juan Campos dos Santos¹, Ayman El-Zoka²

¹Ferrovac AG, Switzerland

²Imperial College London, United Kingdom

Ferrovac pushes the boundaries of how samples can be moved in an ultra-high vacuum (UHV) system to enable atom probe analyses of environment sensitive materials. Today this means moving samples between instruments, labs, and institutes in highly controlled environments. This transfer technology is particularly important for biological samples, electrochemical applications, and other applications that require chemical-structural preservation and contamination prevention. Keeping a controlled sample environment throughout the sample preparation process is a challenge for anyone working with cryogenically sensitive samples. Ice build-up leads to longer processing times in the FIB (focused ion beam), or even to a the loss of the cryogenic state of interest. Our latest developments are to support cryo-UHV transfer from a nitrogen glovebox, through the FIB process, to the analysis in the LEAP. To achieve this we have designed a UHV docking chamber capable of fast loading at cryogenic temperatures. This is coupled with our FerroLoader technology, to allow samples to be transferred from liquid nitrogen to our UHV Cryo Transfer Suitcase (UHVCTS). The key developments here are being able to transfer sample carriers into the UHVCTS while keeping them below the glass transition temperature of amorphous ice. This is crucial for analysis of pristine solid-liquid interfaces. The latest system installed at Imperial College London, with more systems on the way, will provide interesting results regarding the effect this controlled transfer has on measurement results. With these developments, Ferrovac hopes to significantly improve yield through reliable and straightforward sample transfer methods, in ultimately controlled conditions. In further developments, we are extending the transfer capabilities to more model systems including the Leica sample block, the Kratos stub, and flag-style sample plates. Parallel to this work, we are adding new instruments to our network including the new UHV-FIB Nanospace by Tescan and potentially the Apreo 2 SEM by Thermo Fisher.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P4

ATOM PROBE TOMOGRAPHY AND CORRELATIVE WORKFLOWS ENHANCED BY LASERFIB

Gabriel Arcuri¹, Travis Casagrande¹, Ramya Cuduvally¹, Pardis Mohammadpour¹, Brian Langelier¹, Nabil Bassim¹

¹McMaster University, Canada

Connecting atom probe tomography (APT) to analyses at higher length scales and ensuring that sample selection for APT correctly represents the material and features of interest, is a critical factor in the preparation of APT specimens. Focused ion beam (FIB) instruments are a key component of many APT sample preparation procedures. Here, it is shown how the integration of a femtosecond laser ablation module with FIB further expands on these capabilities, and enhances specimen preparation and multi-modal correlative microscopy for APT.

LaserFIB offers a precise and efficient method for the preparation of APT specimens [e.g., 1,2,3], due to the ability of the laser to remove large volumes of material, at rates that are orders of magnitude faster than FIB alone. However, procedures for the identification and targeting of deep internal features using the laser ablation method are not yet well-established. In this work, it is shown how the Zeiss Crossbeam 350 LaserFIB can be utilized in a correlative workflow for site-specific APT specimen preparation from deeply buried regions of interest which are located by 3D X-ray tomography.

We also present other methods by which LaserFIB facilitates APT sample preparation with correlative analysis, including custom tip-holder solutions for enabling easy and robust multi-microscope analysis (e.g., FIB-TKD-TEM-APT).

Overall, this proposed correlative LaserFIB approach to APT sample preparation enables access to sub-surface regions while providing correlative information from centimetre length scales down to atomic structures. Some example applications include studying internal stress corrosion cracking in alloys, internal microelectronic device structures, or the complex internal structure of natural biological and geologic materials.

[1] Rottwinkel, B., Kreuzer, A., Spott, H., Krause, M., Schusser, G., Höche, T. Preparing TEM specimens and atom probe tips by laser machining. *Mircosc. Today*, 2019, 27:40-44.

[2] White, N., Eder, K., Byrnes, J., Cairney, J.M., McCarroll, I.E. Laser ablation sample preparation for atom probe tomography and transmission electron microscopy. *Ultramicroscopy*, 2021, 220:113161

[3] Tkadletz, M., Waldl, H., Schiester, M., Lechner, A., Schusser, G., Krause, M., Schalk, N. Efficient preparation of microtip arrays for atom probe tomography using fs-laser processing. *Ultramicroscopy*, 2023, 246:113672.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P5

AN EXTREME ULTRAVIOLET SOURCE FOR USE ON AN ATOM PROBE TOMOGRAPH

Benjamin W. Caplins¹, Ann N. Chiaramonti¹, Jacob M. Garcia¹, Norman A. Sanford¹, Luis Miaja-Avila¹

¹*National Institute of Standards and Technology, USA*

In this poster, we will describe a coherent extreme ultraviolet (EUV) light source and beamline that is coupled to an atom probe tomography (APT) instrument. The EUV light is generated via a commercially available high harmonic generation system based on a noble gas filled hollow core fiber. The centroid of the EUV spectrum is tunable from around *ca.* 25 eV to 45 eV dependent on the identity of the noble gas in the fiber. The ultrashort EUV pulses (<35 fs) are delivered to the APT analysis chamber via a vacuum beamline that was optimized to maximize photon flux at the APT specimen location while minimizing alignment complexity. We describe the beamline optics including the various tradeoffs involved. Additionally, we characterize the spectrum of the EUV light and how that spectrum evolves as it propagates through the various optical elements. The EUV focus spot size is measured at the APT specimen location using a knife-edge photoemission measurement and the effects of misalignment are simulated and discussed. Potential sources of noise relating to the use of EUV photon energies are discussed with proposed solutions. The long term stability of the instrument is demonstrated over more than a year. Finally, we compare this EUV source and beamline to the previous version, [1] highlighting the relative improvements that were made and how these impact APT measurements.

[1] A. N. Chiaramonti, L. Miaja-Avila, P. T. Blanchard, D. R. Diercks, B. P. Gorman, and N. A. Sanford, "A Three-Dimensional Atom Probe Microscope Incorporating a Wavelength-Tunable Femtosecond-Pulsed Coherent Extreme Ultraviolet Light Source," *MRS Adv.* 4, 2367–2375 (2019).

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P6

INVESTIGATING THE EFFECTS OF SUB-10 K TEMPERATURE OPERATION ON ATOM PROBE TOMOGRAPHY

Alec C. Day¹, Andrew J. Breen², David A. Reinhard³, Thomas F. Kelly¹, Simon P. Ringer²

¹*Steam Instruments, Inc, USA*

²*The University of Sydney, Australia*

³*CAMECA Instruments Inc, USA*

The operating temperature is a critical parameter in atom probe tomography (APT) experiments, affecting spatial precision, mass resolving power, and other aspects of the field-evaporation process. While current commercially available atom probes operate at a minimum temperature of ~25 K, here we present modifications to the mechanical design of the LEAP® and EIKOS™ atom probe systems manufactured by CAMECA® that enable a specimen temperature in the sub-10 K regime [1]. In this study, four materials systems, including pure Al, pure W, doped Si, and GaN, were analyzed using these modified instruments. The effects of conducting atom probe experiments in the sub-10 K regime were assessed using quantitative analysis metrics related to spatial precision, mass resolving power, stoichiometry, and charge-state ratio. Our results show that decreasing the specimen temperature significantly improves spatial precision, allowing the resolution of crystallographic features with higher fidelity [2]. Furthermore, compositional measurements of GaN were more accurate due to favorable changes in the field evaporation process and lower noise floors. The improved spatial precision observed at sub-10 K temperatures is attributed, in part, to the reduced thermal vibrations of the sample atoms. This reduction in thermal energy leads to a decrease in the trajectory spread of evaporated ions, resulting in more accurate spatial information. The effects on mass resolving power are relatively minor, with a slight improvement observed at lower temperatures for certain materials. The stoichiometry and charge-state ratio were found to be insignificantly changed across the temperature range studied. The improved accuracy and resolution observed at sub-10 K temperatures will allow for a better understanding of materials properties and behavior at the atomic scale, leading to new insights into materials design and development. Moreover, the modifications presented in this study provide a new tool for investigating the effects of temperature on the field-evaporation process, opening up opportunities for further investigations of this parameter space.

[1] Day, Alec C., et al. "Exploration of atom probe tomography at sub-10K." *Ultramicroscopy* 241 (2022): 113595. [2] Breen, A. J., et al. "Revealing latent pole and zone line information in atom probe detector maps using crystallographically correlated metrics." *Ultramicroscopy* 243 (2023): 113640.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P7

HIGH QUALITY GALLIUM FREE APT SAMPLE PREPARATION USING PLASMA FIB TECHNOLOGY

Min Wu¹, Chengge Jiao¹, Mikhail Dutka¹

¹*Thermo Fisher Scientific, The Netherlands*

Atom probe tomography (APT) provides microstructural characterization at the highest spatial resolution for 3D compositional analysis of materials. Focused ion beam (FIB) microscopy is an essential technique for orientation and site-specific sample preparation for APT work. Gallium liquid metal ion source (LMIS) technology has been the workhorse for FIB applications since it was introduced commercially more than 30 years ago. Over the years, Ga FIB beam properties and low-acceleration voltage performance have increased steadily. Today's most advanced Ga LIMS FIB probe size is 5 nm to 7 nm of full width at half-maximum, compared to nearly 50 nm in the mid-1980s. Samples prepared from metals and alloy bulk materials based on aluminium, iron, copper and titanium are very sensitive to Ga FIB damage. The damage affects sample surface amorphization, depending on beam energy and incident angle. Damage is also generated by introduced defects such as dislocation loops and formed intermetallic compounds in grain boundaries. In this presentation we will discuss gallium free APT sample preparation using Thermo Scientific™ DualBeam™ system with Xenon plasma FIB (PFIB) technology. We will demonstrate that Xe PFIB is a very promising technique for APT sample preparation with fast throughput for rough milling and better control of APT final polishing using low current density of the Xe ion beam. In addition, since Xe ions originate from a noble gas, they do not react with target materials during APT preparations, therefore Ga contamination is avoided for Ga FIB-sensitive materials. We will also show that using Thermo Scientific™ AutoScript™ 4 or iFast™ Software, it is possible to automate the workflow, starting with the electron beam-protection deposition layer to annular pattern milling and, finally, to low-kV FIB cleaning.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P8

ANALYTICAL CAPABILITY ENHANCEMENT WITH THE INVIZO 6000 WIDE FIELD-OF-VIEW ATOM PROBE

*Yimeng Cheng*¹

¹

Abstract missing

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P9

AUTOMATION ADVANCES IN ATOM PROBE TOMOGRAPHY

Katherine Rice¹, Gard Groth¹, Ty Prosa¹, Yimeng Chen¹, Isabelle Martin¹, Dan Lenz¹, David Reinhard¹

¹CAMECA Instruments Inc.

CAMECA has released a number of automation innovations in recent years to improve throughput and ease-of-use for atom probe tomography. Not only can automation save time and reduce the learning curve for newer users, but the use of automated methods can also improve consistency and repeatability in both data acquisition and data analysis. In this presentation we present the automation advances with current generation instruments such as automated specimen alignment for both LEAP and Invizo, as well as chain acquisition and scripted acquisition implementations. Atom probe acquisition with static experimental conditions can be a largely unattended process, but often the operator wishes to change the data collection conditions throughout the acquisition based on live data feedback, requiring the operator to remain attentive to the process. CAMECA has provided the ability for users to control acquisitions using a graphical user interface-based Script Builder and the ability to upload code written in C# directly in Script Browser. Scripted acquisition has the advantage of being able to change the acquisition conditions to be an optimal match for samples containing more than one type of material, allowing for improved yield and higher data quality. Employing scripted acquisition allows the operator to screen for optimal acquisition conditions for a given sample type and allow live data reporting to circumvent the reconstruction process if desired, for example, concentration can be output to a log file. Data reconstruction can also be automated with the use of a recipe-based reconstruction process to reduce time to knowledge for key APT results. The reconstruction parameters can be changed so that a number of reconstructions and analyses can be launched automatically. The operator can then review the data to select the optimal reconstruction for further analysis or conduct parameter studies which are time consuming in a fully manual workflow. CAMECA is committed to providing tools to improve the atom probe workflow speed, reduce the learning curve and make our users more productive.

Technical Advances in Specimen Preparation and (cyro) Transfer, Instrumentation, Workflow Automation

P10

ADVANTAGES OF SIMULTANEOUS VOLTAGE AND LASER PULSING

Robert M. Ulfiaq¹, Ty J. Prosa¹, Yimeng Chen¹, David A. Reinhard¹, Isabelle Martin¹, Michael Holman¹, Jesse. Robinson¹, Dan Lenz¹, JDavid J. Larson¹

¹CAMECA Instruments Inc.

CAMECA has released a number of automation innovations in recent years to improve throughput and ease-of-use for atom probe tomography. Not only can automation save time and reduce the learning curve for newer users, but the use of automated methods can also improve consistency and repeatability in both data acquisition and data analysis. In this presentation we present the automation advances with current generation instruments such as automated specimen alignment for both LEAP and Invizo, as well as chain acquisition and scripted acquisition implementations. Atom probe acquisition with static experimental conditions can be a largely unattended process, but often the operator wishes to change the data collection conditions throughout the acquisition based on live data feedback, requiring the operator to remain attentive to the process. CAMECA has provided the ability for users to control acquisitions using a graphical user interface-based Script Builder and the ability to upload code written in C# directly in Script Browser. Scripted acquisition has the advantage of being able to change the acquisition conditions to be an optimal match for samples containing more than one type of material, allowing for improved yield and higher data quality. Employing scripted acquisition allows the operator to screen for optimal acquisition conditions for a given sample type and allow live data reporting to circumvent the reconstruction process if desired, for example, concentration can be output to a log file. Data reconstruction can also be automated with the use of a recipe-based reconstruction process to reduce time to knowledge for key APT results. The reconstruction parameters can be changed so that a number of reconstructions and analyses can be launched automatically. The operator can then review the data to select the optimal reconstruction for further analysis or conduct parameter studies which are time consuming in a fully manual workflow. CAMECA is committed to providing tools to improve the atom probe workflow speed, reduce the learning curve and make our users more productive.

Nano-scale characterization of Geological and Planetary Materials

P11

WATER IN QUARTZ SINGLE CRYSTALS: AN ATOM PROBE TOMOGRAPHY STUDY

Luiz F. G. Morales¹, Stephan Gerstl¹

¹ETH Zürich, Switzerland

Silicon dioxide, also known as silica or quartz, is a prevalent mineral found in the Earth's crust and potentially on other planets. It has piqued interest due to its chemical and physical stability and diverse polymorphs that make it suitable for various industrial and technological applications. The mechanical behavior and chemical stability of quartz are crucial to comprehend in natural and experimental conditions, and the study of hydrous components in anhydrous phases like quartz has been a subject of study for decades. While infrared spectroscopy is the preferred method to characterize hydrous components in minerals due to its sensitivity to OH components, it remains uncertain how these molecules are distributed and stored in quartz. To visualize the distribution and nature of water species in quartz, we conducted Atom Probe Tomography (APT) on an oriented sample of a quartz single crystal. The quartz single crystal was obtained from the Schinschlucht area in Graubünden, Switzerland, cut along the (0001) plane, mounted in epoxy, and polished for EBSD. The SEM-cathodoluminescence imaging indicated a homogenous sample with no fractures or inclusions, and the EBSD analysis confirmed the orientation of the section ($\pm 2^\circ$), with a negligible amount of lattice distortion. From this sample, we prepared five oriented APT tips via Ga-FIB, parallel to the [0001] quartz direction, with their apex ranging from 100 to 150 nm in diameter. We successfully collected two ~ 1.106 atom datasets from two of these tips, one in voltage-pulsing mode and one in laser-pulsing mode. Both had similar counting statistics, and the first analyses indicated the presence of both molecular water and OH heterogeneously distributed along the analyzed volume. Our initial results indicate that hydrous components in quartz single crystals are not homogeneously distributed, and we plan to perform element frequency distribution, cluster analysis, and charge-state comparisons on both datasets.

Nano-scale characterization of Geological and Planetary Materials

P12

EXPLORATORY APPLICATION OF APT-BASED CORRELATIVE MICROSCOPY TO LUMINESCENT ZIRCONS

Florant Exertier¹, Jonathan Houard¹, Martin Bizzarro², Rick Verberne², Lorenzo Rigutti¹

¹*University of Rouen Normandie, France*

²*University of Copenhagen, Denmark*

The application of Atom Probe Tomography (APT) to geological materials has recently disclosed new possibilities into the nanoscale characterization of these systems, yielding important results in the interpretation of the processes that characterize their growth and the subsequent modifications [1, 2]. APT may indeed be exploited because of its 3D nanoscale resolution within a multiscale approach including a set of microscopical and microanalytical techniques. As a particular class of phenomena, optical emissions in minerals are mostly related to the presence of impurities and defects within a matrix, and this can be the object of a correlative nanoscale characterization targeting both chemical – possibly structural – and optical properties. In this framework, cathodoluminescence (CL) may be applied as a spatially and spectrally resolved technique [3]. In this contribution we explore the possibility of correlating the information obtainable by APT with that obtainable by optical spectroscopy, in particular within the recently developed Photonic Atom Probe (PAP) [4]. As a model system, we analyze zircons ($ZrSiO_4$) sampled from Jack Hills. Zircon is a robust geochronometer and one of the most widely used minerals across the geosciences to quantify the nature, timing, and duration of geological processes. Zircon exhibits a strong CL signal and a significant optical emission with spatially varying luminescence properties, also due to the presence of luminescent quartz inclusion within the zircon matrix. Our exploratory results indicate the possibility to obtain different types of correlation at different scales: (a) The correlation of CL or energy-dispersive X-ray (EDX) hyperspectral maps with the APT analysis of tips whose location is controlled on the crystal surface (b) The correlation of optical PL spectra and 3D APT reconstructions within the in-situ approach based on the PAP. The significance and perspectives of these correlative approaches for geoscience will be discussed.

[1] J.W. Valley et al. *Nature Geoscience*, 7,214 (2014)

[2] S. M. Reddy et al. *Geostandards and Geoanalytical Research*, 44,1, p. 5-50, (2020)

[3] J. Götze, *Microscopy and Microanalysis* 18, p. 1270–1284, (2012)

[4] J. Houard et al. *Review of Scientific Instruments* 91.8, p. 083704, (2020)

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P13

PARAMETER STUDY OF ATOM PROBE TOMOGRAPHY OF 304 STAINLESS STEEL USING LEAP 6000 XR

David Mayweg¹, Janne Schilling¹, Isac Johansson², Simon Dovrén², Lukas Palmgren¹, Mattias Thuvander¹

¹*Chalmers University of Technology, Sweden*

The instrument LEAP 6000 XR (Cameca) has a laser with wavelength 258 nm, which is shorter than previous versions of commercial instruments. The instrument is equipped with a reflectron for energy compensation, and the instrument has an expected detection efficiency of 52%. Here we have performed a parameter study on 304 stainless steel, to foremost investigate the influence of laser pulse energy on mass resolution, mass spectrum background, charge-state distribution, multiple events and surface diffusion. The effect of pulse fraction on the performance in voltage pulse mode has also been investigated. Furthermore, the effect of using simultaneous laser and voltage pulsing is investigated. The main conclusions are that the mass resolution remains very good also at laser pulse energies large enough to cause surface diffusion of silicon, and that the background can be suppressed by using simultaneous laser and voltage pulsing.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P14

REVEALING CRYSTALLOGRAPHIC INFORMATION IN SILICON FROM FIELD DESORPTION IMAGES

Jeroen E. Scheerder¹, Yngwie Baron^{1,2}, Richard J.H. Morris¹, Claudia Fleischmann^{1,3}

¹IMEC, Belgium

²Eindhoven University of Technology, The Netherlands

³KU Leuven, Belgium

Poles and zone lines, typically observed as fluctuations in the hit density histogram, are related to the crystallography of the specimen. This information can aid in improving the atom probe tomography reconstruction. For instance, the identification of multiple poles allows to determine the image compression factor [1]. Unfortunately, these features are not always readily available from the hit density histogram. In Si<001> pre-sharpened microtips on the LEAP 5000XR, solely the (004) pole can be identified this way. In this work, we improve on the crystallographic information obtained in Si<001>. This was achieved by filtering the detector event histogram based on the number of pulses between detection events N_p at a specific detector location. This parameter has been shown to correlate with crystallography, as there can be a local reduction of N_p at specific sites such as low-index poles [2]. Two methods which have been shown to reveal additional information are (i) computing the average number of pulses between detection events per pixel [3], and (ii) filtering the detector event histogram by only including detection events with N_p below a certain threshold t , $N_{p<t}(x,y)$ [3,4]. We show that for acquisitions performed in voltage mode, method (i) is successful in revealing additional poles such as the <311> family in silicon. For the same data, we introduce a novel extension of method (ii) to reveal additional crystallographic information. By computing the fraction of hits satisfying $N_{p<10}(x,y)$ per pixel rather than the absolute amount, more poles as well as other four-fold symmetric features become visible. For the analysis of the specimen in laser mode, not all the same poles can be identified, though (other) four-fold symmetric features appear. Possible origins for the observed features and the difference between voltage and laser mode are discussed, as well as a case study where the filters are used to track the influence of experimental parameters on the image compression factor for Si<001>.

[1] B. Gault *et al.*, *Microsc. Microanal.* 14, 296 (2008)

[2] L. Yao *et al.*, *Appl. Phys. Lett.* 107, 241602 (2015)

[3] L. Yao *et al.*, *MethodsX* 3, 268 (2016)

[4] A.J. Breen *et al.*, *Ultramicroscopy* 243, 113640 (2023)

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P15

MAGNIFICATION ARTEFACTS OF CORE-SHELL EMITTERS DEPENDENT ON ACQUISITION CONDITIONS IN APT

Lorenzo Rigutti¹, Eric Weikum¹, Enrico Di Russo¹, Georges Beainy¹, Pradip Dalapati¹, Jean-Michel Chauveau², Maxime Hugues³, Denis Lefebvre³, Jonathan Houard¹, Angela Vella¹, François Vurpillot¹

¹University of Rouen Normandie, France

²Université Versailles St Quentin/Paris Saclay, France

³Université Côte d'Azur

The Photonic Atom Probe (PAP) allows for simultaneous collection of photoluminescence (PL) spectra from a field emission tip specimen and for its analysis through field evaporation as in standard laser-assisted atom probe tomography (APT) [1]. Besides the interest of this instrument as a microscope [2], the particular conditions in which the optical signatures of localized light emitters are collected open intriguing possibilities for the study of field ion emission under high field and under laser illumination [3]. As an example, the PL spectral shift allows measuring the stress induced by the application of a strong electric field at the tip apex and its propagation through the tip. This has been evidenced both through the study of the stress-induced splitting of the zero-phonon line of the NV⁰ center in diamond [4] and of the quantum well (QW) emission in a ZnO/(Mg,Zn)O system [5], allowing measuring stress levels ranging from 9 GPa to ~1 GPa. Time-resolved measurements evidence the growing role of non-radiative recombination as the tip surface progressively approach a localized emitter. Furthermore, it can be shown that the evolution of the tip shape and the accumulation of free carriers at the tip surface may have an effect on the absorption pattern of the laser light within the tip and on the far-field pattern of the emitted PL [6].

[1] J. Houard *et al.*, *Review of Scientific Instruments*, vol. 91, no. 8, p. 083704, Aug. 2020, doi: 10.1063/5.0012359.

[2] E. Di Russo *et al.*, *Nano Lett.*, vol. 20, no. 12, pp. 8733–8738, Dec. 2020, doi: 10.1021/acs.nanolett.0c03584.

[3] E. Di Russo and L. Rigutti, *MRS Bulletin*, vol. 47, no. 7, pp. 727–735, Jul. 2022, doi: 10.1557/s43577-022-00367-6.

[4] L. Rigutti *et al.*, *Nano Lett.*, vol. 17, no. 12, pp. 7401–7409, Dec. 2017, doi: 10.1021/acs.nanolett.7b03222.

[5] P. Dalapati *et al.*, , doi: 10.1103/PhysRevApplied.15.024014.

[6] E. Weikum, "Physical mechanisms influencing the far field photoluminescence emission of an evaporating field emission tip," *Accepted in the J. of the Optical Society of America A*.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P16

EXPLORING FIELD-ASSISTED NITROGEN ACTIVATION WITH ATOM PROBE MICROSCOPY

Sten V. Lambeets¹, Mark G. Wirth¹, Daniel E. Perea¹

¹Pacific Northwest National Laboratory, USA

In addition of its role as fertilizer, NH₃ is considered as a promising mean of energy storage. To unlock this potential, it is critical to lower energy requirements and generate a zero-carbon NH₃ by coupling with renewable electricity¹. Promising results have been demonstrated with Single Atom electroCatalysts (SAeCs) with local High External Electric Fields (HEEFs) to dissociate N₂. If those effects present promising outcomes according to theoretical calculation^{2,3}, the values of those HEEFs and the mechanisms involved remains largely unknown and unexplored. We will present and demonstrate how Field Ion Microscopy (FIM) and Operando Atom Probe (OAP) are ideal techniques to unravel those mechanisms at the nanoscale since they inherently rely on HEEFs for imaging process. This demonstration will be illustrated with the first results of our investigation of room temperature N₂ dissociation over Ru. We use the recently developed OAP technique⁴ to measure this dissociation over a 0001-oriented Ru specimen. After fixing a constant HEEF between 15 and 25V/nm and the temperature at 300K, 1.4x10⁻⁷mbar N₂ pure gas is introduced in the analytic chamber. N₂⁺(28 Da), N₂²⁺ and/or N⁺(14 Da), and N²⁺(7 Da) species have been detected. Nⁿ⁺ are mainly detected over the Ru{1012} facets while N₂ⁿ⁺ are detected on large areas in the periphery of the imaged apex. FIM and OAP are capable to observe and estimate the HEEF necessary to trigger specific chemical reaction steps such as the N₂ dissociative adsorption (i.e. activation). With an accurate calculation of those HEEFs, those values can be extrapolated to create new chemical and reactor system designed to perform N₂ activation at relatively low energy cost. In a context of electrification of chemical processes, OAP can help pave the way to a deeper understanding of the physical laws involved in electrochemistry, as well as in chemistry in general.

1.M.Wan *et al.* Deep Learning-Assisted Investigation of Electric Field–Dipole Effects on Catalytic Ammonia Synthesis. *JACS Au* 2,1338–1349(2022).

2.S.M.Kathmann, Electric fields and potentials in condensed phases. *Phys.Chem.Chem.Phys.* 23,23836–23849(2021).

3.M.L.Karahka & H.J.Kreuzer, New physics and chemistry in high electrostatic fields. *Surf.Sci.* 643,164–171(2016).

4.S.V.Lambeets *et al.* Nanoscale Perspectives of Metal Degradation via In Situ Atom Probe Tomography. *Top.Catal.* 1606-1622(2020)

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P17

FIELD EMISSION PHYSICS WITHIN A PHOTONIC ATOM PROBE

Nadya Spettel¹, Jeroen E. Scheerder², Tsung-Wei Chou¹, Yuan Tu², Shreya Garg¹, Bo Zhao³, Christophe Detavernier³, Claudia Fleischmann^{1,2}, André Vantomme¹

¹KU Leuven, Belgium

²IMEC, Belgium

³Ghent University, Belgium

The size of devices in the semiconductor industry is decreasing while their complexity is increasing, making atom probe tomography (APT) a promising technique due to its nanoscale 3D compositional analysis capabilities. However, the ambiguous results of oxide analyses in APT demand additional fundamental research before the technique reaches its full potential [1]. In this study, we perform APT on silicon pre-sharpened microtips (PSM) conformally coated with thin oxide layers by atomic layer deposition (VO₂, amorphous VO_x, indium tin oxide (ITO)). The use of PSM's circumvents ion-beam milling induced effects like Ga-implantation or amorphization of the outer layer and allows us to, e.g., study the laser-tip interaction more easily. We found that upon changing the acquisition conditions the magnification of the silicon core on the detector alters significantly. While similar observations have been reported on core-shell field emitters (GaP|Si and GaP|SiGe in [2], Si|ZnO in [3]), a full explanation is lacking. To close this gap, we determined the apex shape after APT measurements with atomic force microscopy using a novel tip-to-tip alignment method [4]. A non-hemispherical apex depending on the acquisition conditions was observed, with a convex (concave) silicon core corresponding to a high (low) magnification as was observed at low (high) laser powers at constant detection rate. With 2D evaporation simulations the experimental apex shapes were reproduced semi-quantitatively by varying the evaporation probabilities of the constituent materials and the link between apex shape and magnification could be confirmed. The key parameter for this behaviour was found to be the applied voltage (hence the field) in the experiments. From these findings we conclude that the relative evaporation probability of the core compared to the shell might change significantly, i.e., the Si core has a higher (lower) evaporation field compared to the oxide at high (low) applied fields and we discuss different factors as potential root-causes.

[1] A. D. Giddings *et al.*, *Scripta Materialia* 148 (2018): 82-90.

[2] S. Koelling *et al.*, *ECS Transactions* 75.8 (2016): 751.

[3] Z. Sun *et al.*, *Ultramicroscopy* 184 (2018): 225-233.

[4] J. Op de Beeck *et al.*, *The Journal of Physical Chemistry C* 124.11 (2019): 6371-6378.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P18

ENERGETIC MAPPING IN ATOM PROBE TOMOGRAPHY USING FIELD EVAPORATION ENERGY LOSS SPECTROSCOPY

Vurpillot François¹

¹*University of Rouen Normandie, France*

Atom Probe Tomography (APT) is a technique used to map the 3D composition of a specimen at the near atomic scale for a wide range of materials. However, no real local chemical information is usually extracted from the tomographic data, which requires intensive post-treatments coupled with APT datasets and simulation models. In the past, APT data have been indirectly mined by studying the mechanism of field evaporation of surface atoms. It is known that Field evaporation has shown differences from one sample to another, which are related to the atomic local environment and can help explain their properties. In a recent study, we have shown that energetic considerations in the field evaporation process are present within the data but have been ignored until now [1]. Mass spectra shapes using voltage pulse analyses are related to the activation energy required by the field evaporation mechanism, which can be used to extract local energetic information with nanometric resolution. The approach is termed "Field Evaporation Energy Loss Spectroscopy" (FEELS) and can be adapted to any straight flight path atom probe instrument using voltage pulses, even on existing data sets. We demonstrate how this information can be mapped in 1D, 2D, and 3D dimensions in a few experimental cases. This information reveals fine-scale energetic considerations in the field evaporation process, which depends on the material and analysis temperature and is universal to the material nature and atomic organization. An example in a pure Al dataset is presented in the paper.

[1] "Introducing field evaporation energy loss spectroscopy", Loïc Rousseau et al., Communications Physics, in press

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P19

FIELD EMISSION PHYSICS WITHIN A PHOTONIC ATOM PROBE

Jan Erik Rybak¹, Tim M. Schwarz², Baptiste Gault^{2,3}, Cynthia A. Volkert¹

¹*Georg-August-Universität Göttingen GmbH, Germany*

²*Max-Planck-Institut für Eisenforschung GmbH, Germany*

³*Imperial College London, United Kingdom*

Specimen fracture is one of the major obstacles that stands in the way of successful atom probe tomography (APT) of brittle materials. A variety of different explanations for fracture have been put forward, based on the idea that it initiates where the mechanical stresses induced by the high electric fields are largest. Although certain experimental trends have been explained by these arguments, there is large scatter in the observations. A mechanistic and predictive understanding has not yet been achieved, probably in part because of the sensitivity of fracture to nanoscale defects and tip geometry. In an attempt to obtain a clear picture of the conditions leading to brittle fracture of APT specimens, dedicated experiments have been performed on a wide range of silicon tips prepared by focused ion beam (FIB) milling or lithographically defined and etched microtip arrays (Cameca PSM M36). We vary doping, crystallographic orientation, treatment with ion and electron beams, and number and nature of defects in the Si. We introduced notches on the side of the specimens by means of FIB machining, to produce known stress concentrations. The tips are then tested in a custom build laser assisted atom probe or a voltage pulsed LEAP 5000 XS, where the DC electric field is increased at a steady rate until the specimen fails or the maximum electric potential is reached. First tests on more than 50 specimens show failure either almost immediately, before controlled field evaporation of Si has set in, or they show stable field evaporation and never fail. Lithographically defined tips have a 100% chance of early fracture after imaging with electrons or ions, while un-imaged tips never fail. FIB machined tips containing Li fail immediately, while those without Li never fail, independent of their doping state. Neither the FIB-machining method nor the presence of notches is found to have any detectable effect on fracture. The observed trends point to some yet unidentified parameter(s), that have a strong effect on fracture. Further tests should provide a decipherable path to the underlying causes of fracture by APT and will be presented at the conference.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P20

FIELD EVAPORATION THEORY: COMMENTS ON THE BASIC PHYSICS OF BONDING ENERGY AND FIELD EVAPORATION ACTIVATION ENERGY

Richard G. Forbes¹

¹University of Surrey, United Kingdom

This presentation re-examines, at a basic conceptual level, and in a more detailed way than previously, two basic energy-like quantities involved in field evaporation (FEV) theory: bonding energy and activation energy. For simplicity, only the case of single atoms being removed from metallic-element emitters is discussed. Bonding energy is equivalently defined either as: (1) the work done in removing an atom from a specified location on a surface of specified geometry to remote field-free space; or (2) the difference between total system energies before and after atom removal (considering all relevant electrical effects). For neutral solid bodies, the following are discussed. (*1) "Sublimation energy" as work needed per atom to dis-assemble a large crystal. (*2) The 1920s Kossel-Stranski TLK (Terrace-Ledge-Kink-site) model that—for a simple cubic crystal—deduces the bonding energy of a kink-site atom as equal to the sublimation energy. (*3) Arguments, using conservation of energy, suggesting validity of this result for all large crystallographic structures (as traditional FEV theory assumes). (*4) The idea that any "local effects" "move along one" when a kink-site atom is removed. For bonding in electrostatic fields, the following are discussed. (*5) The different types of electrostatic field that may be relevant, and problems in calibrating field values, and the theoretical advantages of field ratios. (*6) The different mechanisms by which electrostatic fields can affect bonding energy. (*7) The historical discussion that concluded that fields F increase kink-site bonding energy by $\epsilon_0 \Omega F^2 / 2$, where Ω is atomic volume. (*8) The possible roles of field-induced crystallographic strain energy and density functional theory (DFT). In respect of FEV activation energy $Q(F)$, the following issues are discussed. (*9) The "seven-dimensional" FEV flux equation. (*10) The different $Q(F)$ regimes in traditional ionization-determined FEV theory, and related formulae. (*11) The "quasi-thermodynamic" concept of partial (activation) energy, and the different types thereof, and historical experimental values. (*12) Relationship of partial energies with the "C-parameter" of modern AP theory. (*13) Alternative physical interpretations, within traditional theory, of $Q(F)$ and partial energies. (*14) The role of DFT theory in traditional FEV theory, and the advisability of scientific validation. (*15) The need for careful scientific discussion of proposed force-based FEV theories. Finally, the presentation identifies issues needing further research.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P21

TUTORIAL REMARKS ABOUT USING THE SCHOTTKY CONSTANT IN FIELD ION EMISSION THEORY

Richard G. Forbes¹

¹University of Surrey, United Kingdom

This Poster creates greater commonality in basic high-field theory affecting potential-energy (PE) barriers in electron emission and gas field ionization, and also the basic thermodynamic formula for zero-barrier (ZB) evaporation field. The Schottky constant (c here, often c_s elsewhere) is a universal constant given (to 7 sig. fig.) in the International System of Quantities by $c \equiv (e^3/4\pi\epsilon_0)^{1/2} \cong 1.199985 \text{ eV V}^{-1/2} \text{ nm}^{1/2} = 3.794686 \times 10^{-5} \text{ eV V}^{-1/2} \text{ m}^{1/2}$, with e the elementary positive charge. This Poster shows c appears in each context above. Initial algebraic formulation of ideas behind c was Schottky's in 1914. The name is modern. For simplicity, only basic cases are discussed. (1) Consider electron emission from a free-electron metal with a planar surface of large lateral extent, with image forces. Let an electron approaching the emitter surface normally from inside, with no applied field, see a PE barrier of zero-field height H . Applying an electrostatic field of magnitude F reduces the classical barrier height by energy $\Delta = cF^{1/2}$. This is the *Schottky effect*. The ZB field-magnitude that reduces the classical barrier height to zero is $F_{ZB}(e^-) = c^{-2}H^2$. (2) Consider field ionization (FI) of a hydrogenic ion with nuclear charge Ze (i.e, a single electron orbits charge Ze) and with ionization energy I_Z . In one-dimensional theory (disregarding field-induced changes in I_Z), the ZB field-magnitude that reduces the classical barrier to electron escape to zero is $F_{ZB}(FI-Ze) = 4c^{-2}(I_Z)^2$. The factor "4" relates to the difference between a Coulomb PE and an image PE. (3) The well-known basic thermodynamic formula for the ZB escape field for field evaporation into an escape charge-state with ion charge ne is $F_{ZB}(FEV-ne) = n^{-3}c^{-2}(K_n)^2$, where K_n is the thermodynamic term that also defines the *local* zero-field activation energy Q_{0n} . The Poster indicates how formulae are derived. For (1) and (2), the ZB fields define the *scaled field* f corresponding to applied field F , by $f=F/F_{ZB}$. This f is used in JWKB-type tunnelling theory, as applied to field electron emission (FE) and FI, that involves the FI/FE special mathematical function $v(f)$.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P22

USING FOWLER-NORDHEIM PLOTS TO "MEASURE" CHARACTERISTIC LOCAL FIELD VALUES

Richard G. Forbes¹

¹University of Surrey, United Kingdom

This Poster is part of a planned project to improve some basic scientific aspects of atom-probe theory. Difficulties in making reliable absolute comparisons between theory and experiment in field ionization and field evaporation (and also field electron emission) are in reliably defining and measuring the experimental electrostatic field. For field ion emission, the traditional solution (not fully precise) has been: (a) define a reliable "reference situation", most commonly best image conditions for helium ion imaging of a (110)-oriented tungsten emitter of moderate apex radius, near emitter temperature 80 K; (b) establish, by some "defined calibration procedure" what value of a "specified field quantity" (e.g., the apex field in the critical surface) corresponds to the reference situation (the value usually taken is 44 V/nm or the rounded value 45 V/nm); and (c) establish by applied-voltage comparisons, and by assuming field is proportional to applied voltage, what value of the specified field-quantity (or some well-defined related field-quantity) applies to the phenomenon of interest, for example field evaporation onset under specified conditions. Several choices of "defined calibration procedure" exist. The original choice involved the theory of field electron emission; a later choice involved field ionization experiments above best image voltage; more recently, methods involving in-situ emission in electron microscopes have been proposed; possibly methods involving atomic-level electrostatic calculations will eventually become available. The last 20 years have seen significant advances in the theory of field electron emission (FE). This Poster has a tutorial function and is intended to provide background information about a modern method of estimating experimental field-magnitudes in the context of FE, with some examples. In modern FE theory the *scaled field* $f = F/F_R$ where F is the local surface field-magnitude and F_R is the F -value needed to reduce to zero (for a Fermi-level electron) a Schottky-Nordheim barrier of zero-field height equal to the local work-function. The method works by extracting f -values from Fowler-Nordheim plots of experimental FE current-voltage data, and using these to determine F -values and the relationship between F and applied voltage V . The Poster explains the method. An earlier version can be seen at: <https://doi.org/10.13140/RG.2.2.13837.77280>.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P23

ROLE OF IMPURITIES IN FIELD ION MICROSCOPY CONTRAST

Shalini Bhatt¹, Shyam Katnagallu¹, Christoph Freysoldt¹, Jörg Neugebauer¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

Specimen fracture is one of the major obstacles that stands in the way of successful atom probe tomography (APT) of brittle materials. A variety of different explanations for fracture have been put forward, based on the idea that it initiates where the mechanical stresses induced by the high electric fields are largest. Although certain experimental trends have been explained by these arguments, there is large scatter in the observations. A mechanistic and predictive understanding has not yet been achieved, probably in part because of the sensitivity of fracture to nanoscale defects and tip geometry. In an attempt to obtain a clear picture of the conditions leading to brittle fracture of APT specimens, dedicated experiments have been performed on a wide range of silicon tips prepared by focused ion beam (FIB) milling or lithographically defined and etched microtip arrays (Cameca PSM M36). We vary doping, crystallographic orientation, treatment with ion and electron beams, and number and nature of defects in the Si. We introduced notches on the side of the specimens by means of FIB machining, to produce known stress concentrations. The tips are then tested in a custom build laser assisted atom probe or a voltage pulsed LEAP 5000 XS, where the DC electric field is increased at a steady rate until the specimen fails or the maximum electric potential is reached. First tests on more than 50 specimens show failure either almost immediately, before controlled field evaporation of Si has set in, or they show stable field evaporation and never fail. Lithographically defined tips have a 100% chance of early fracture after imaging with electrons or ions, while un-imaged tips never fail. FIB machined tips containing Li fail immediately, while those without Li never fail, independent of their doping state. Neither the FIB-machining method nor the presence of notches is found to have any detectable effect on fracture. The observed trends point to some yet unidentified parameter(s), that have a strong effect on fracture. Further tests should provide a decipherable path to the underlying causes of fracture by APT and will be presented at the conference.

Fundamental aspects in APT, Laser-Matter Interaction, high-field Nanoscience, Field Ion Microscopy, Simulations

P24

UNDERSTANDING LITHIUM'S FIELD EVAPORATION BEHAVIOR FROM FIRST PRINCIPLES

Shyam Katnaqallu¹, Huan Zhao¹, Se-Ho Kim¹, Jörg Neugebauer¹, Baptiste Gault¹, Christoph Freysoldt¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

Lithium (Li) is a critical metal for battery operation due to its high energy density, low weight, and high mobility. To develop efficient batteries, atomic-scale characterization of complex, Li-containing materials is crucial. Atom probe tomography (APT) could provide extremely valuable insights, however, APT faces many evaporation artefacts that can render the data unusable, particularly for pure Li [1]. In this study, we used density functional theory (DFT) that explicitly includes high electric fields [2] to investigate Li's field evaporation behaviour. Our results show that Li's field evaporation behaviour arises from its preference for adsorption at on-top sites on a Li (110) surface, which results in a reduction of energy compared to adsorption on a hollow/bridge site. We systematically study the surface-diffusion of Li adatoms as a function of the electric field and discovered the existence of a "critical" electric field below the field evaporation strength of Li, where the on-top site becomes energetically unfavourable. This leads to a barrier-less diffusion of Li atoms on the surface, which explains the spotty evaporation pattern observed experimentally. To prevent this undesired effect, we explored potential approaches to minimize surface diffusion before field evaporation. One approach involves depositing a monolayer of gas such as H, N and He, onto the Li surface. We show, that such an adsorbed gas layer prevents Li atoms from diffusing on the surface.

[1] S. H. Kim, S. Antonov, X. Zhou, L. T. Stephenson, C. Jung, A. A. El-Zoka, D. K. Schreiber, M. Conroy, and B. Gault, *Atom Probe Analysis of Electrode Materials for Li-Ion Batteries: Challenges and Ways Forward*, J. Mater. Chem. A 10, 4926 (2022).

[2] C. Freysoldt, A. Mishra, M. Ashton, and J. Neugebauer, *Generalized Dipole Correction for Charged Surfaces in the Repeated-Slab Approach*, Phys. Rev. B 102, 45403 (2020).

Nano-scale characterization of Semiconductor Materials and Devices

P25

INHOMOGENEITY OF RANDOM BINARY ALLOYS AND THEIR COUNTING STATISTICS IN ATOM PROBE TOMOGRAPHY

Masoud Dialameh¹, Jeroen E. Scheerder¹, Claudia Fleischmann^{1,2}

¹*KU Leuven, Belgium*

²*IMEC, Belgium*

Semiconductor alloys are increasingly being utilized in the industry due to their adjustable optical and electrical properties, which outperform intrinsic semiconductors. These properties can be tailored through various processes, ranging from standard doping up to development of complex functional alloys [1]. The optoelectrical characteristics of alloys are primarily governed by their composition and the atomic-scale distribution of elements. In the case of random alloys, the elemental distribution is known to follow a binomial distribution [2]. Consequently, inherent statistical inhomogeneity exists within small nanometric volumes. Atom Probe Tomography (APT) has proven to be a potent technique for investigating the elemental distribution in semiconductor alloys, providing depth and spatial resolution at the nanometer scale [3]. However, limited knowledge exists regarding the uncertainty of APT compositional analysis for random alloys, as well as its limitations in probing small volumes. In this study, we have developed an analytical expression that enables the calculation of the statistical inhomogeneity in random binary alloys, taking into account the voxel size and average composition based on the binomial distribution. This analytical expression provides insights into the inherent inhomogeneity found within small nanometric volumes of random binary alloys. Additionally, the counting statistics of Atom Probe Tomography (APT) were modelled using a separate binomial distribution, which was then combined with the intrinsic inhomogeneity of random binary alloys. By integrating these two distributions, we have established an analytical expression capable of predicting the theoretical uncertainty of APT compositional analysis, considering factors such as voxel size, average composition, and detector efficiency. Lastly, we have compared our theoretical model with the experimentally obtained uncertainty derived from the APT data.

[1] K. Kaneko et al., *J Appl Phys* 113, 233901 (2013).

[2] M. P. Moody et al., *Microsc Res Tech* 71, 542 (2008).

[3] L. Rigutti, et al., *J Appl Phys* 119 (2016).

Nano-scale characterization of Semiconductor Materials and Devices

P26

SODIUM IN CU(IN,GA)SE₂ SOLAR CELLS: TO BE OR NOT TO BE BENEFICIAL

Azam Karami^{1,2}, Marcin Morawski³, Heiko Kempa³, Roland Scheer³, Oana Cojocaru-Mirédin^{1,2}

¹RWTH Aachen University, Germany

²Albert-Ludwigs Universität Freiburg, Germany

³Institution Martin-Luther-Universität Halle-Wittenberg, Germany

The Cu(In,Ga)Se₂ thin-film polycrystalline absorbers contain many grain boundaries (GBs), which were believed to be electrically detrimental for the cell performance, although, the record efficiency of the polycrystalline absorbers confirm their superiority to the monocrystalline counterparts. The cause is mainly attributed to the passivation effect at GBs by incorporation of alkali dopants in the CIGS absorber. Na doping have exhibited a drastic improve of cell efficiency, the V_{OC} and fill factor (FF). On the contrary, Na doping in too high extend will lead to immediate degradation of the cell performance. Therefore, the main question is in which extend Na must be incorporated to reach an optimum performance? In the present work, different techniques such as electron backscatter diffraction (EBSD), atom probe tomography (APT) and admittance spectroscopy are used to investigate the effect of different dosages of Na doping on the cell performance. It shows that the moderate addition of Na strongly decreases the V_{OC} deficit, however, further addition of Na to a higher content increases the V_{OC} deficit and showing a higher density of GBs, dislocations and precipitates. Donor defects at GBs are not detrimental since it attracts electrons and the electrons can easily channel through the GBs and collect to the front contact. But donor defects within clusters trap the electrons or sink them. Since they are found in the bulk material and they are not connected to any grain boundaries, the electrons remain trapped and they are not any more freely moving inside the CIGS absorber layer. Thus, the voltage measured will be much lowered, the conversion efficiency will be decreased and finally, the solar cell performance will be degraded. In conclusion, this work provides for the first-time, clear statements about the detrimental effect of Na when found in very high quantity.

Nano-scale characterization of Semiconductor Materials and Devices

P27

INHOMOGENEITY OF RANDOM BINARY ALLOYS AND THEIR COUNTING STATISTICS IN ATOM PROBE TOMOGRAPHY

Azam Karami^{1,2}, Semih Agca³, Heiko Kempa³, Roland Scheer³, Oana Cojocaru-Mirédin^{1,2}

¹*RWTH Aachen University, Germany*

²*Albert-Ludwigs Universität Freiburg, Germany*

³*Institution Martin-Luther-Universität Halle-Wittenberg, Germany*

Tandem cell development is now an ongoing venture in both research and industry. The new generation of tandem solar cells based on Cu(In,Ga)Se₂ (CIGS) technology requires the development of higher band gap CIGS absorber. The Cu-rich CIGS material is characterized by a higher band gap than the standard Cu-poor CIGS absorber. Although, due to the poor performance of the cells with Cu-rich CIGS absorber, Ag alloying into the absorber layer has become a vital solution. However, the Ag solubility and redistribution inside CIGS absorber is still unknown. In this work, we employed the electron backscatter diffraction (EBSD) and atom probe tomography (APT) techniques in order to determine the Ag distribution inside the CIGS absorber layer and its possible effect on grain boundaries (GBs) properties, when inserted under low, medium and very high content. The results show that the fluctuations in composition of the absorber layer appears when $AAC = \frac{[Ag]}{[Ag]+[Cu]}$ is greater than 0.2. These chemical fluctuations discussed in this work are related to possible spinodal decomposition effects.

Nano-scale characterization of Semiconductor Materials and Devices

P28

TRANSPARENT NIOBIUM-DOPED TITANIUM DIOXIDE THIN FILMS BY REACTIVE MAGNETRON SPUTTERING FOR THERMOELECTRIC MODULES

Joana M. Ribeiro¹, Alexander Welle², Torben Boll², Carlos J. Tavares¹

¹University of Minho, Portugal

²Karlsruhe Institute of Technology, Germany

Transparent thermoelectric materials are a promising technology for touchscreen displays and solar cell applications, rendering a sustainable powering of the device. Modifying the atomic structures of TiO₂ by deliberately introducing defects can enhance its electrical and thermoelectrical properties to a great extent. This work reports the production and characterization of optically transparent Nb-doped TiO₂ thin films with enhanced thermoelectric properties deposited by reactive d.c. magnetron sputtering in high vacuum. The optimization of the production parameters results in thin films with thickness of approximately 200 nm, maximum average optical transmittance in the visible range of 76 %, n-type electrical resistivity of 0.01 Ω·cm and a maximum absolute Seebeck coefficient of 226 μV·K⁻¹, resulting in a maximum thermoelectric power factor of 300 μW·K⁻²·m⁻¹. Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) and Atom probe tomography (APT) are powerful tools to understand the role of Nb doping, oxygen flow, deposition temperature and annealing in the properties of the thin films, enabling a further fine tuning of the production method. APT analyses enabled the determination of the composition and inherent homogeneity, as well as to investigate the cation and anion segregation to interfaces and grain boundaries.

Nano-scale characterization of Semiconductor Materials and Devices

P29

ANALYSIS OF SCALN EPITAXIAL LAYERS BY ATOM PROBE TOMOGRAPHY

Samba Ndiaye¹, Aïssatou Diagne¹, Caroline Elias², Heélène Rotella², Frédéric Georgi³, Maxime Hugues², Yvon Cordier², François Vurpillot¹, Lorenzo Rigutti¹

¹University of Rouen Normandie, France

²Université Côte d'Azur, France

³PSL Research University, France

Due to its large piezoelectric and spontaneous polarization coefficients the wide bandgap ScAlN alloy is becoming a promising material in III-nitride semiconductor technology, as high-frequency surface and bulk acoustic wave devices but also the next generation of high electron mobility transistors (HEMTs). The epitaxial growth of ScAlN has been recently developed by few groups but it is in its infancy and necessitates in-depth structural studies. Laser-assisted Atom Probe Tomography (La-APT) was applied to the study of thin $\text{Sc}_x\text{Al}_{1-x}\text{N}$ layers grown by molecular beam epitaxy with ammonia source as nitrogen precursor. The structural properties of the thin ScAlN barriers have been carefully studied using X-ray diffraction and reflectivity, X-ray photoelectron spectroscopy (XPS), and secondary ion mass spectrometry (SIMS). Field evaporation takes place at fields that can be estimated as slightly higher than 23 V/nm, a value close to the typical values for AlN [1]. The measured III-site alloy fraction within the $\text{Sc}_x\text{Al}_{1-x}\text{N}$ layer is $x=0.11$, which is lower than the Sc concentration determined from XPS (14.5%) and SIMS (14%) measurements. The origin of this difference is under investigation. The Sc fraction is approximately constant along the depth coordinate, confirming the Sc profile extracted from XPS measurements using in-situ argon ions etching. However, it exhibits a slight dependence on the surface field, similarly to what can be found in other III-N alloys, x decreases with increasing field[2]. This may point to an increase of the loss of Sc with increasing electric field, with Sc being more prone to evaporation than Al. Furthermore, the alloy distribution of Sc was investigated. The Sc-Sc first nearest neighbor analysis indicated that the alloy does not exhibit clustering and rather behaves as a random alloy[3]. In summary, these analyses indicate the applicability of APT to the study of ScAlN, but also the need for a more in-depth investigation of compositional metrology in this alloy.

[1] L. Mancini *et al.*, *J. Phys. Chem. C*, doi: 10.1021/jp5071264.

[2] E. Di Russo *et al.*, *J. Phys. Chem. C*, doi: 10.1021/acs.jpcc.8b03223.

[3] L. Rigutti *et al.*, *Journal of Applied Physics*, doi: 10.1063/1.4943612.

Nano-scale characterization of Semiconductor Materials and Devices

P30

ANALYSIS OF SCALN EPITAXIAL LAYERS BY ATOM PROBE TOMOGRAPHY

Samba Ndiaye¹, Enrico Di Russo², Sébastien Duguay², François Vurpillot¹, Chiara Carraro², Gianluigi Maggioni², Enrico Napolitani², Lorenzo Rigutti¹

¹University of Rouen Normandie, France

²Università degli Studi di Padova, Italy

The capability of Atom Probe Tomography (APT) to perform micro-structural analyses has been recently extended to the study the effect of a large number of semiconductors manufacturing processes including ion implantation and rapid thermal processes (e.g., flash lamp annealing, laser annealing...) [1]. Among them, pulsed laser melting (PLM) was recently emerged not only as a reliable method to activate doping during the fast recrystallization occurring after the melting regime. Recently, a novel approach based on PLM on Ge has been developed in order to create box-like alloys with controlled thickness and composition on scales of a few hundred nanometers, with a good control of defect formation and excellent dopants activation levels. All these intriguing features - that are peculiar of PLM - have been recently exploited to fabricate Ge:Sn with record active concentrations [2]. On the other hand, producing heavily doped Ge:Sb layer with conventional epitaxy techniques remains a big challenge due to the low solubility of group-V materials in Ge [3]. Within this context, APT is a unique tool for boosting the development of heavily doped materials, combining 3D atomic-scale imaging capabilities, single atom sensitivity and analytical capability. In this contribution, we report APT analyses performed on Ge:Sn and Ge:Sb layers, in which we assess species distribution and possible clustering to extended defects.

[1] V. Heera *et al.*, "Depth-resolved transport measurements and atom-probe tomography of heterogeneous, superconducting Ge:Ga films," *Supercond. Sci. Technol.*, vol. 27, no. 5, p. 055025, Mar. 2014, doi: 10.1088/0953-2048/27/5/055025.

[2] E. Di Russo *et al.*, "Fully relaxed Ge_{0.9}Sn_{0.1}/Ge by Sn/Ge deposition and nanosecond pulsed laser melting," *Submitted*.

[3] T. K. P. Luong, V. L. Thanh, A. Ghrib, M. E. Kurdi, and P. Boucaud, "Making germanium, an indirect band gap semiconductor, suitable for light-emitting devices*," *Adv. Nat. Sci: Nanosci. Nanotechnol.*, vol. 6, no. 1, p. 015013, Jan. 2015, doi: 10.1088/2043-6262/6/1/015013.

Nano-scale characterization of Semiconductor Materials and Devices

P31

CORRELATIVE MICROSCOPY WITHIN A PHOTONIC ATOM PROBE

Lorenzo Rigutti¹, Jonathan Houard¹, Enrico Di Russo¹, Georges Beainy¹, Pradip Dalapati¹, Jean-Michel Chauveau², Maxime Hugues³, Denis Lefebvre¹, Ioanna Dimkou⁴, Eva Monroy⁴

¹University of Rouen Normandie, France

²Université Versailles St Quentin/Paris Saclay, France

³Université Côte d'Azur, France

⁴Université Grenoble-Alpes, France

The laser pulses controlling the ion evaporation in Laser-assisted Atom Probe Tomography (La-APT) can simultaneously excite photoluminescence (PL) in semiconductor or insulating specimens. An atom probe equipped with appropriate focalization and collection optics can thus be coupled with an in-situ micro-PL bench [1] that can be operated even during APT analysis. Such a Photonic Atom Probe (PAP) has been applied to the study of the optical properties of nanoscale emitters in an in-situ correlative microscopy approach. The PAP discloses new possibilities for the correlative microscopy of nanoscale light emitters, as the evolution of the PL spectra can be directly correlated with the chemical 3D distribution obtained by APT. As a result, PAP can distinguish the optical signatures of separate QWs distant as few as 20 nm – well below the diffraction limit of the laser [2]. It can furthermore be applied to the study of quantum dots, in order to assess the relationship between their 3D morphology and their optical signature[3], and to the study of the interplay between the presence of structural or point defects and the possibility of radiative recombination of carriers [4]. So far, II-oxide and III-nitride heterostructures have been studied with this technique. In perspective, it is possible to consider its application to the study of heterostructures in other semiconducting systems but also of emitting defects in semiconducting and in geologic materials.

[1] J. Houard *et al.*, "A photonic atom probe coupling 3D atomic scale analysis with in situ photoluminescence spectroscopy," *Review of Scientific Instruments*, vol. 91, no. 8, p. 083704, Aug. 2020, doi: 10.1063/5.0012359.

[2] E. Di Russo *et al.*, "Super-resolution Optical Spectroscopy of Nanoscale Emitters within a Photonic Atom Probe," *Nano Lett.*, vol. 20, no. 12, pp. 8733–8738, Dec. 2020, doi: 10.1021/acs.nanolett.0c03584. [3] I. Dimkou *et al.*, "InGaN Quantum Dots Studied by Correlative Microscopy Techniques for Enhanced Light-Emitting Diodes," *ACS Appl. Nano Mater.*, vol. 3, no. 10, pp. 10133–10143, Oct. 2020, doi: 10.1021/acsanm.0c02106.

[4] Ioanna Dimkou, "Luminescence from InGaN/GaN quantum dots correlating in-situ micro-photoluminescence and atom probe tomography: Effect of structural defects and point defects.," *Accepted in Microscopy and Microanalysis*, 2023.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P32

TRIBOLOGICALLY-INDUCED OXIDATION OF A ZIRCONIUM NITRIDE MULTILAYER COATED KNEE EXPLANT

Julia Rau¹, Andrea Fazi¹, Mattias Thuvander¹

¹*Chalmers University of Technology, Sweden*

Among the increasing number of artificial joint replacements, total knee arthroplasty (TKA) is widely spread [1]. These joints are subjected to friction and wear due to the complex interplay of tribological loading from the human body environment and motion. The surfaces of the joint components – femoral and tibial component and a polyethylene articulating surface in between – may exhibit chemical reactions and microstructural changes. Among these changes, an often-observed phenomenon in a wide range of sliding contacts is tribologically-induced oxidation. Oxidized implant surfaces were observed for zirconium nitride covered implants after explanting them from the human body. For patients with a metal allergy, the implants' CoCr₂₉Mo₆ base material is coated with an up to 7 μm thick multilayer (alternating layers of CrN and CrCN and a top-most ZrN layer) [2]. In this study, an explant taken from a 72-year-old male patient (102 kg) after 2 years due to aseptic loosening [3] is investigated with Atom Probe Tomography (APT) with particular interest in the oxide containing surface layer formed. In addition, a fresh implant will be used for comparison. The results of APT will be discussed in terms of degradation mechanism of the implants' coating. In the worst-case, oxide formation could lead to wear particle formation within the joint and potentially to implant failure. Hence, understanding the fundamental mechanisms of this phenomenon is of utmost importance extending the lifetime of TKA.

[1] Statistisches Bundesamt, Fallpauschalenbezogene Krankenhausstatistik, 2020, 2021.

[2] J. Reich, L. Hovy, H.L. Lindenmaier, R. Zeller, J. Schwiesau, P. Thomas, T.M. Grupp, *Orthopaede*, 2010, 39, 495–502.

[3] A. L. Reyna, J. Lützner, B. Altermann, T.M. Grupp, *International Case Reports Journal*, 2023, 3, 1–16.

[4] A.L. Reyna, B. Fritz, J. Schwiesau, C. Schilling, B. Summer, P. Thomas, T.M. Grupp, *J Biomech.*, 2018, 79, 88–96.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P33

CAN ATOM PROBE TOMOGRAPHY REVEAL MICROSTRUCTURAL DETAILS IN LITHIUM INSERTED CARBON FIBRES?

Marcus Johansen¹, Mahander Pratap Singh², Baptiste Gault^{2,3}, Fang Liu¹

¹Chalmers University of Technology, Sweden

²Max-Planck-Institut für Eisenforschung GmbH, Germany

³Imperial College London, United Kingdom

Lithium-ion batteries play a key role in electrification of transportation, thus will contribute greatly to a more sustainable future. To further reduce the weight of electric vehicles on the system level, a novel multifunctional battery – structural battery composite – was proposed and recently showed promising multifunctional performance [1]. The device carries load (like a conventional carbon fibre reinforced composite) and store electrochemical energy simultaneously. Thus, carbon fibres function both as reinforcements and negative electrode. The microstructure of Li inserted carbon fibres still remains largely unknown. Recently, Johansen et. al. used Auger electron spectroscopy (AES) to analyse lithiated carbon fibres and found relatively uniform distribution of Li in carbon fibres [2]. However, with a spatial resolution larger than 10 nm, AES cannot reveal the details on the atomic level. Our recent study used APT to analyse pristine carbon fibres and resolved the complex mass spectra [3]. It laid the foundation to take one step further and use atom probe tomography (APT) to reveal the microstructural details in lithiated carbon fibres. There are a still several big challenges for the use of APT on Li inserted carbon fibres: first, lithiated carbon fibres are sensitive to ambient air; second, the material tend to fracture pre-maturely during the analysis; third, the composition of the lithiated carbon fibres and its proximate regions are complex. In this study, we reveal the effects of specimen transfer – air transfer, chromium coating combined with air transfer, and vacuum transfer; we compare the effects of different instruments – with either reflectron or straight flight path – on the background level and mass resolution of the mass spectra. Finally, we shed some light on the field evaporation phenomenon of Li inserted carbon fibres, particularly emphasise on the behaviour of molecular ions. The results pave the way to employing APT to analyse (re)active carbonaceous electrode materials.

[1] L. E. Asp, et. al. *Advanced Energy and Sustainability Research*, p 2000093, 2021.

[2] M. Johansen, et. al. *Applied Surface Science*, 2023.

[3] M. Johansen, et. al. *Carbon*, Vol. 179, p 20–27, 2021.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P34

PHASE TRANSFORMATIONS OF SNSBGBITE ALLOY ANODE PARTICLES IN LITHIUM-ION BATTERY

Andrea Fazi¹, Sajid Alvi¹, Patrik Johansson¹, Mattias Thuvander¹

¹*Chalmers University of Technology, Sweden*

Lithium-ion batteries (LIBs) are widely used for various applications, including electric vehicles, consumer electronics, and grid storage. Recent studies have investigated the use of alloy anode, such as silicon, germanium, and tin, as an alternative to graphite to increase the energy density in LIBs ¹. Alloy anodes offer advantages such as higher energy density, faster charging times, and longer battery life. However they can also experience significant volume changes during (de)lithiation, leading to pulverization and loss of capacity over time. To address this issue, researchers are developing new electrode materials with improved stability and lifespan using various strategies, such as nano-engineering and composites ^{2,3}. High entropy alloys (HEAs) are a type of alloy containing five or more principal elements in roughly equal proportions, offering unique properties ⁴. HEAs can improve the mechanical stability of the alloy anode during (de)lithiation due to solid solution strengthening. In this work, the chemical stability of SnSbGeBiTe HEA as an anode material in LIB was investigated using atom probe tomography (APT). APT measurements of SnSbGeBiTe particles are taken in their as-produced state, and after one and three battery cycles. In the as-produced material, Ge-O rich particles of 2-5 nm in size are uniformly distributed in a SnSbGeBiTe matrix. After the first three cycles, the SnSbGeBiTe phase is found to be enriched with 5-10 at.% lithium left behind during the de-lithiation step. Ge-O particles remain unaffected by the battery cycle, and Li appears to be captured by the interface between these particles and the matrix. In certain regions, a Sb-rich phase can be found to have segregated rejecting most of the other alloying elements. No additional change is observed after three cycles. These results can explain the loss of capacity observed in the electrical measurements after the first cycle and the subsequent stabilization of the system.

[1] Wang, X., *et al.*, Mater. Today 50, 259–275 (2021).

[2] Zheng, P. *et al.* Batter. Supercaps 202200481, (2022).

[3] Guo, M. *et al.* Front. Chem. 10, 1–13 (2022).

[4] Miracle, D. B. *et al.*, Acta Mater. 122, 448–511 (2017).

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P35

INVESTIGATING THE SECONDARY CORROSION PROTECTION OF FE-BASE ALLOYS BY MEANS OF ATOM PROBE TOMOGRAPHY

Amanda Persdotter¹

¹Chalmers University of Technology, Sweden

The challenge of high temperature corrosion is often addressed by the use of highly alloyed materials such as stainless steels or FeCrAl alloys. The corrosion protection of these alloys rely on the formation of a protective Cr- and/or Al- rich corundum type oxide. However, in harshly corrosive environments, these oxides tend to break down resulting in the formation of a fast-growing Fe-rich oxide scale, a process known as breakaway corrosion. Systematic studies on the growth mechanisms of the Fe-oxides formed after breakaway are scarce. However, recent studies have shown that the growth rate of the Fe-rich oxide may be significantly reduced by the change in alloy composition (see e.g. [1]). The Fe-rich oxide scales are in general multi-layered, composed of an outward-growing pure Fe-oxide and an inward-growing mixed oxide scale. It has been proposed that the inward-growing scale is the most critical part in determining the corrosion protection after breakaway, i.e. the secondary corrosion protection. The microstructure and chemical composition of this part of the oxide-scale is complex, containing pores and oxides with nano-sized sub-grains. The large amount of sub-grain boundaries would suggest that grain boundary diffusion through the inward-growing oxide may play an important role for the growth mechanism of these oxide scales. Thus, this study utilizes the high resolution and three-dimensional analysis of atom probe tomography to give insight into how the distribution of alloying elements within the inward-growing oxide scale may influence the protection against corrosive species such as chlorine and alkali. The study investigates the oxide scales formed on four Fe-based model alloys chosen to exhibit good (Fe₁₀Cr₄Al₂Si, Fe₁₈Cr₁₀Ni) as well as poor (Fe₁₀Cr₄Al, Fe₁₈Cr₂Ni) secondary corrosion protection. The oxides are thermally grown for 168 hours at 600 °C in a corrosive environment containing 5%O₂+20%H₂O+N₂+500 vppm HCl(g) + 1 mg/cm² KCl(s). The results have the potential to greatly aid in the understanding of high temperature corrosion for many applications where breakaway corrosion remains a challenge.

[1] Persdotter et al., Beyond Breakaway corrosion - Influence of chromium, nickel and aluminum on corrosion of iron-based alloys at 600 °C, Corrosion Science 177, (2020)

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P36

PREPARATION OF LOW-MELTING SCALMS PARTICLES FOR ATOM PROBE MEASUREMENTS

Nora Vorlaufer¹, Simon Carl¹, Erdmann Spiecker¹, Peter Felfer¹

¹Friedrich-Alexander-University Erlangen-Nürnberg, Germany

Supported Catalytically Active Liquid Metal Solution (SCALMS) are a new catalyst type, which is used e.g. in dehydrogenation reactions [1,2]. SCALMS show promising catalytical properties with a high selectivity and a low deactivation. The particles consist of a low melting matrix, like Ga, which is doped with a catalytic active material, like Pt. To investigate the chemical composition of SCALMS, we use APT. APT proved to be suitable to reveal detailed information about the particle chemistry [3,4]. Due to the low solidus temperature of the Ga-particles of around 30 °C [5], the APT specimen preparation is carried out at cryo-temperatures. In this presentation we introduce a workflow that allows us to incorporate SCALMS particles inside of an APT specimen. To achieve this, we attach the particles to a substrate tip using dielectrophoresis and create a void-free particle-matrix compound using electrodeposition. This preparation approach leads to a near-tip shaped geometry, with only minor FIB milling required to shape a final tip. Especially the cryo-preparation in the FIB is done without the need of a cryo-FIB lift-out or a cryo-transfer. In general, the approach itself is simple and allows for a high specimen throughput. By combining STEM EDS measurements and the APT results of these specimens, we draw a precise picture of the chemical distribution of the SCALMS particles. The inner part of the particle comprises pure Ga, while the outer part consists of a heterogenous shell. The shell consists of a Ga-oxide layer, on top of which a pure Pt-layer formed.

[1] N.Taccardi et al., Nat. Chem., 2017

[2] N.Raman et al., ACS Catal., 2021

[3] Felfer et al., Angew. Chem. Int. Ed., 2014

[4] Kim et al., Ultramicroscopy, 2018

[5] R.Meyers, Encyclopedia of physical science and technology, Elsevier Science, 2001

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P37

ENHANCING THE ATOM PROBING OF BATTERY MATERIALS WITH IMPROVED SAMPLE PREPARATION TECHNIQUES

Mahander Pratap Singh¹, Eric V. Woods¹, Se-Ho Kim¹, Baptiste Gault^{1,2}

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

²Imperial College London, United Kingdom

Rechargeable Li-ion batteries (LIBs) will be central for future green energy demands. However, their performance and reliability need to be improved further. Atom probe tomography (APT) with a unique combination of high spatial resolution and elemental sensitivity offers opportunities to study the materials used in LIBs, including cathode, anode, and electrolytes, and gain insights into the nanoscale chemistry and their microstructure changes both in a pristine state and after cycling. However, atom probe studies of Li-containing compounds have been challenging due to several factors. The preferential evaporation of Li under high electric field (delithiation), compositional variation due to pulsed laser applied during APT are prominent of them. Preparing sharp needle-shaped APT specimens of such beam-sensitive samples adds further challenges including Ga-implantation, Ga damage, and phase amorphization. In this presentation, I will discuss two studies on Li-containing materials. First, by adopting a cryogenic specimen preparation technique, we evaluated the surface chemistry of the evolved passive layer that forms when $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ reacts with ambient air [1]. Second, show how in-situ coating of APT specimens can significantly improve our data quality and reduce the failure rate during APT analysis, and significantly counteract the Li-migration under a high electric field [2]. These developments further advance the atom probe studies for challenging materials, especially batteries, providing better insights into the atomic-scale behavior of the notorious Li-ions.

[1] M.P. Singh, S.-H. Kim, X. Zhou, H. Kwak, A. Kwiatkowski da Silva, S. Antonov, L.S. Aota, C. Jung, Y.S. Jung, B. Gault, *Advanced Energy and Sustainability Research* 4 (2023) 2200121.

[2] E.V. Woods, M.P. Singh, S.-H. Kim, T.M. Schwarz, J.O. Douglas, A. El-Zoka, F. Giuliani, B. Gault, arXiv:2303.18048 (2023).

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P38

FACILITATING ATOM PROBE TOMOGRAPHY OF FREE-STANDING 2D MXENE FILMS

Mathias Krämer¹, Bar Favelukis², Maxim Sokol², Brian A. Rosen², Noam Eliaz², Se-Ho Kim³, Baptiste Gault^{1,2}

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

²Tel Aviv University, Israel

³Korea University, Republic of Korea

⁴Imperial College London, United Kingdom

2D materials, including MXenes, are promising nanomaterials that may outperform commercial materials in applications such as energy storage and catalysis. During wet chemical synthesis of the MXenes, the 2D transition metal carbides and nitrides are terminated with a variety of functional groups, and cations such as Li⁺ are used to intercalate into the structure to obtain delaminated single nanosheets. The variety of elements used in the synthesis therefore makes it necessary to determine the detailed chemical composition, in order to better assess and understand the relationships between composition and properties of these materials. To prepare MXene electrodes, the material ink from synthesis is vacuum filtered through a membrane to obtain an additive-free, free-standing film. In this contribution, we show how to perform atom probe tomography of these films through the use of a recently introduced in-situ coating approach in the focused ion beam [1]. This allows for the analysis of much larger volumes of the 2D nanomaterial with atom probe tomography compared to other workarounds [2], thus enabling more advanced statistical data analysis. Since various 2D material inks can be processed into free-standing films, the presented approach could also be an enabler for the atom probe analysis of other 2D materials.

[1] Woods, Eric V., et al. "A versatile and reproducible cryo-sample preparation methodology for atom probe studies." arXiv preprint arXiv:2303.18048 (2023)

[2] Krämer, Mathias, et al. "Near-Atomic Scale Perspective on the Oxidation of Ti₃C₂T_x MXenes: Insights from Atom Probe Tomography." *In Preparation* (2023)

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P39

IDENTIFYING MULTIELEMENT ACTIVE SITES AS A DESIGN STRATEGY FOR HIGH-PERFORMANCE LASER-GENERATED H₂O₂ ELECTROCATALYSTS

Varatharaja Nallathambi¹, Cui Wang², Sven Reichenberger³, Baptiste Gault¹, Alexander Eychmüller², Stephan Barcikowski³, Dierk Raabe¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

²Technische Universität Dresden, Germany

³University of Duisburg-Essen, Germany

In search of economically sustainable catalysts as a replacement for noble metals, recent research has shown promising potential in transition metal-based multicomponent systems. High entropy alloys (HEA) or compositionally complex alloys (CCA) popularly known for their attractive mechanical properties have gained widespread attention in the field of catalysis latterly. The scalable synthesis and availability of durable electrocatalytically-active CCA nanoparticles with desired composition as well as crystalline or amorphous structure from pulsed laser ablation in liquids have been shown in the past [1]. Up until now, particularly the structural and chemical complexity of these alloys serves as a tool in designing and optimizing their surface chemical behavior, making them an emerging alloy playground for understanding and modulating catalytically active sites. However, the wide compositional space makes it difficult to understand and correlate the structure-chemical-activity relationships, the shortcomings of nanoparticle size effects, and the need for support structures during catalytic testing. To bridge this knowledge gap, the present work implements a combinative approach between pulsed-laser ablation in liquids (PLAL) and the field of gelation to produce (CoCrFeMnNi) CCA nanoparticles-based aerogels whose compositional distribution is characterized to atomic length scales by atom probe tomography (APT) in correlation with their respective activity as heterogeneous electrocatalysts [2]. It is found that the laser-generated and gelated quinary (CoCrFeMnNi) CCA aerogels exhibit excellent performance in 2e⁻ oxygen reduction reaction (ORR) with a H₂O₂ selectivity of over 90%. To gain insights into the element-dependant surface active site combinations that favor the electrocatalytic H₂O₂ formation, three exemplary quaternary systems without Co, Fe and Ni respectively were synthesized and electrocatalytically evaluated. A combinative spectro-chemical analysis using APT and X-ray photoelectron spectroscopy (XPS) was attempted to unravel the pronounced activity trends between the respective surface compositions. Overall, the presented study highlights the utilization of APT to characterize complex alloys at atomic length scales aiding the design strategies for developing sustainable electrocatalysts.

[1] J. Johnny, et al., Laser-generated high entropy metallic glass nanoparticles as bifunctional electrocatalysts. *Nano Research*, 15(6): p. 4807-4819, (2021).

[2] A. Eychmüller, Nanoparticle-Based Aerogels and Their Prospective Future Applications. *The Journal of Physical Chemistry C*, 126(45): p. 19011-19023, (2022).

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P40

ROLE OF CA DOPING IN THE ACTIVITY AND STABILITY OF LACOO₃ NANOPARTICLE ELECTROCATALYSTS TOWARDS OXYGEN EVOLUTION REACTION

Fan Bai¹, Tong Li¹

¹*Ruhr University Bochum, Germany*

Electrocatalytic water splitting is a key technology to produce hydrogen to meet the ever-increasing demand for sustainable energy. However, increasing the efficiency and longevity of water electrolyser remains a notorious challenge due to the limitations in activity and stability of anode electrocatalysts at which oxygen evolution reaction (OER) occurs.[1] LaCoO₃ (LCO) has been regarded as a promising candidate due to its superior OER activity.[2] However, LCO exhibited poor OER stability.[3] Doping LCO with Ca is an effective way to improve stability and activity, but the effects of Ca remain elusive. This study aims to elucidate how Ca doping improves the activity and stability of LCO and why LCO degrades rapidly. Here, LCO and Ca-doped LaCoO₃ nanoparticles (LCO-Ca-0.4) were synthesized by hydrothermal method. The overpotential of LCO-Ca-0.4 is ~372 mV at 10 mA cm⁻², which is lower than that of LCO (416 mV), indicating that LCO-Ca-0.4 has higher OER activity than LCO. After 1000 cyclic voltammetry (CV) cycles, the OER activity of LCO decreases remarkably since the overpotential increases to 525 mV. In contrast, there is a negligible change in the OER activity of LCO-Ca-0.4, demonstrating that Ca doping effectively improve the OER stability of LCO-Ca-0.4. To further explore the role of Ca in the stability, LCO and LCO-Ca-0.4 were characterized by high-resolution transmission electron microscopy (HRTEM) and atom probe tomography (APT). We observe by HRTEM amorphous layers of 5 - 10 nm on the surface of LCO after 1000 CV cycles, while LCO-Ca-0.4 remains crystalline after OER. Additionally, APT data reveals that the surface of LCO is lean in La, suggesting the surface amorphization is mainly induced by La leaching. In contrast, the surface concentration of LCO-Ca-0.4 remains unchanged, which implies that Ca doping most likely restrains the La leaching. This work demonstrates that the stability of LCO can be improved by Ca doping since Ca could prevent La leaching and inhibit surface amorphization during OER.

[1] Y. Jiao et al. Chemical Society Reviews, 2015, 44, 2060-2086.

[2] C. W. Sun et al. Advanced Energy Materials, 2021, 11, 2000459.

[3] R. Xie et al. ACS Applied Energy Materials, 2021, 4, 9057-9065.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P41

PIEZORESPONSE ENHANCEMENT IN PT-DOPED WO₃ THIN FILMS

Pamela M Pineda Domínguez¹

¹Universidad Autónoma de Ciudad Juárez, Mexico

WO₃ is a *n*-type semiconductor with outstanding chromogenic and photoelectrocatalytic properties with applications in gas sensors and electrochromic device fabrication. WO₃ thin films also have potential as an active layer in water splitting processes for “green” hydrogen¹. At the present, lead-free nano-ferroelectric materials have recently been attracting attention in water splitting technology for their piezocatalytic properties², based on the mechanism of converting mechanical into chemical energy. Therefore, the present work focuses on the piezoelectric properties of WO₃ thin films with and without Pt doping with a thickness range of 200 nm – 250 nm. The films were deposited by sputtering at 225 W of RF power from commercial WO₃ targets (99.95%) and heat-treated post-deposition at temperatures ranging from 400 °C to 600 °C under a forming gas atmosphere (3% H₂ in N₂)³. Switching spectroscopy piezoresponse force microscopy (SS-PFM) shows a piezoresponse enhancement in the Pt-doped films. Structural characterization carried out by X-ray diffraction (XRD) and transmission electron microscopy (TEM) reveals a mixture of phases providing a local non-symmetry related to piezo active domains. Atom probe tomography (APT) was used to study the effects of platinum doping on WO₃ grain boundaries and nanovoid distribution inside the films.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P42

ATOM PROBE TOMOGRAPHY ANALYSIS OF BaFe_2As_2 SUPERCONDUCTORS

Laura Lain Rodriguez¹, Shah Alam Limon², Chiara Tarantini², Eric Hellstrom², Fumitake Kametani², Chris Grovenor¹, Michael Moody¹, Susie Speller¹

¹University of Oxford, United Kingdom

²Florida State University, USA

The microstructure and chemistry of materials plays a big role in their potential superconducting properties, as demonstrated by the artificial introduction of defects in the widely commercially used NbTi, or the careful control of the oxygen content in cuprate superconductors. Iron-based superconductors (IBS), a family of superconductors found in 2006¹, is no exception to this, with several studies highlighting the negative impact that nanoscale variations in chemistry and impurities at grain boundaries can have on the superconducting current^{2,3}. Within the IBS, BaFe_2As_2 (or 122-type) materials have a range of benefits with respect to other types of superconductor, including cost-effective manufacturing, low electromagnetic anisotropies, a critical temperature of up to 38 K⁴, and most importantly their high critical current densities and fields⁵. 122 materials could therefore be suitable for industries seeking higher critical current densities maintained through large magnetic fields, for applications such as medical imaging, magnetic energy storage systems, particle accelerators and fusion reactors. However, challenges remain still to optimise their current-carrying capabilities if they are to be used in polycrystalline form industrially. Because of the almost atomic resolution provided by Atom Probe Tomography (APT) and its capability to study the structure of materials in three-dimensions⁶, it is an ideal technique to investigate the nanoscale chemistry variations within 122 compounds. This poster will therefore focus on initial findings of APT analysis for the study of nanoscale characteristics of BaFe_2As_2 materials as part of a project on improving the processing and properties for future applications.

[1] Kamihara et al., 'Iron-Based Layered Superconductor: LaOFeP '.

[2] Kim et al., 'Evidence for Composition Variations and Impurity Segregation at Grain Boundaries in High Current-Density Polycrystalline K- and Co-Doped BaFe_2As_2 Superconductors'

[3] Pak et al., 'Synthesis Routes to Eliminate Oxide Impurity Segregation and Their Influence on Intergrain Connectivity in K-Doped BaFe_2As_2 Polycrystalline Bulks'

[4] Rotter, Tegel, and Johrendt, 'Superconductivity at 38 K in the Iron Arsenide ($\text{Ba}_{1-x}\text{K}_x$) Fe_2As_2 '.

[5] Tarantini et al., 'Significant Enhancement of Upper Critical Fields by Doping and Strain in Iron-Based Superconductors'

[6] Gault, *Atom Probe Microscopy*.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P43

STRUCTURE-PROPERTY RELATIONSHIPS OF GRAIN BOUNDARIES IN PBTE USING CORRELATIVE TECHNIQUES

Oana Cojocaru-Mirédin^{1,2}, Riga Wu², Yuan Yu², Matthias Wuttig²

¹University of Freiburg, Germany

²RWTH Aachen University, Germany

Grain boundaries (GBs) play a significant role in controlling the transport of mass, heat and charge. However, no clear demonstration of the mechanism underpinning the charge carrier scattering at GBs have been given until now due to the difficulty to determine the local properties of internal interfaces. Therefore, in this work we will demonstrate that correlative techniques, consisting of a combination of the atom probe tomography with electron backscatter diffraction and Hall effect setup (for local transport measurements), can be applied to determine local properties of grain boundaries in PbTe. For the PbTe material, the strength of carrier scattering at GBs depends on its misorientation angle and composition. A concomitant change in the barrier height is observed, significantly increasing from low- to high-angle GBs. Moreover, atom probe tomography measurements reveal that Ag decoration changes strongly between the low-angle grain boundaries (Ag enrichment at dislocations cores) and high angle grain boundaries (a compact Ag layer enrichment) explaining the strong change in charge mobility observed. The compositional and structural changes are accompanied by a breakdown of the dielectric screening, which explains the enlarged GB barrier height. These findings correlate charge carrier scattering with composition locally, promising new avenues for the design of advanced functional materials.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P44

ANALYSIS OF THE OXIDE FORMATION ON PD CATALYSTS DURING NO OXIDATION BY ATOM PROBE TOMOGRAPHY

Yoonhee Lee¹, Daniel Dobesch¹, Patrick Stender¹, Ute Tuttlies¹, Ulrich Nieken¹, Guido Schmitz¹

¹University of Stuttgart, Germany

To investigate the surface modifications of palladium catalyst during NO conversion reactions, a combined study using a Flat Bed Reactor (FBR), Temperature-Programmed Reduction (TPR) and Atom Probe Tomography (APT) was performed. The Palladium (Pd) catalysts reveal a decreased conversion rate after the first heating cycle of the FBR measurement, which is attributed to the formation of an oxide layer on top of the metallic catalyst surface. The total oxygen content adsorbed on the surface of the catalyst was determined by TPR for catalyst particles that were produced by the spark discharge method. To analyse oxidation microscopically, Pd and, for comparison, Pt and PtPd tips of comparable curvature radius were treated analogously to the FBR processing and investigated by APT. Using a custom-made furnace attached to the APT system, the cycling process of the catalyst particles was replicated and the effective oxide thicknesses were determined. During the 1st heating cycle, the degree of oxidation increases and once the temperature reaches 350°C, an irreversible layer of PdO oxide of 1.5 nm in thickness is formed. No preference for particular facets of the crystalline Pd surface could be determined.

Nano-scale characterization of Metals and Alloys

P45

EFFECT OF NATURAL AGEING AND CU CONTENT ON THE SOLUTE CLUSTERS AND PRECIPITATES FORMED IN HIGH-STRENGTH 6XXX

James Famelton¹, Ceri Williams², Carla Barbatti¹, Paul Bagot³, Michael Moody³

¹*Brunel University London, United Kingdom*

²*Innoval Technology Ltd, United Kingdom*

³*University of Oxford, United Kingdom*

Reducing automotive vehicle is crucial for improving vehicle energy efficiency and the drive towards electrification. Al-6XXX alloys offer a cost-effective combination of strength, ease of manufacture and good recyclability. Further improvements in strength can lead to further weight reductions and the adoption of these alloys for more automotive components. It is well known that natural ageing of 6XXX alloys may be detrimental to subsequent ageing and is thought to be related to the formation of solute clusters [1-3]. Copper addition to an alloy has been suggested to reduce the deleterious effect of natural ageing [4]. Atom probe tomography (APT) is unique in its ability to observe the solute clusters that form during natural ageing and pre-ageing of these alloys, providing both spatial and chemical information at the very early stages of precipitation. Prototype extrusions of a low Cu content alloy and a high Cu variant were made, both with and without a short period of natural ageing, to observe the separate and combined effects of these two variables. The microstructural characterisation of the samples has been carried out using APT at multiple stages of the ageing process. A study of the influence of natural ageing time and copper content on composition, size distribution, number density and morphology of solute clusters and precipitates will be presented. The techniques and analysis methods used to extract this information from the APT data will also be discussed.

[1] S. Pogatscher et. al, Acta Mater., vol. 59, no. 9, pp. 3352–3363, 2011.

[2] G. H. Tao et. al, Mater. Sci. Eng. A, vol. 642, pp. 241–248, 2015.

[3] A. Poznak et. al, Mater. Sci. Eng. A, vol. 721, pp. 47–60, 2018.

[4] S. Wenner et. Al, Int. J. Mater. Res., vol. 103, no. 8, pp. 948–954, 2012.

Nano-scale characterization of Metals and Alloys

P46

COMBINED MICROSCOPIC STUDIES ON THE NANOPRECIPITATES EVOLUTION IN AN ADDITIVELY MANUFACTURED MARAGING STEEL

Sadhasivam Murali¹, Konda Gokuldoss Pradeep¹

¹*ITT-Madras, India*

Maraging steels are precipitation strengthened steels characterized by a superior combination of ultra-high strength and good fracture toughness. This unique combination of mechanical properties in maraging steels is due to the nano-scale intermetallic precipitate formation in the BCC matrix during ageing treatment. This presentation, focuses on the evolution of two specific types of nanoprecipitates namely, Ni-Ti type and Fe-Mo type as a function of ageing temperature and time in an additively manufactured 300 grade maraging steel. A combined multiscale microstructural characterization involving Scanning Electron Microscopy (SEM)/Electron Backscattered Diffraction (EBSD)/ Energy Dispersive X-ray Spectroscopy (EDS) at bulk scale and Transmission Electron Microscopy (TEM)/Transmission Kikuchi Diffraction (TKD)/Atom Probe Tomography (APT) at nano/near-atomic length scale was employed to study the microstructural evolution, crystallographic/structural information, and chemistry of nanoprecipitates. APT analysis revealed the coarsening of both Ni-Ti and Fe-Mo based precipitates with increasing ageing temperature (from 510 °C to 540°C) and time (from 4h to 8hr at each temperature). APT further confirms the presence of Fe-Mo type precipitate in globular morphology while the Ni-Ti based precipitate in elongated disc-like morphology. The same has been corroborated with the TEM and STEM imaging along with correlative elemental mapping. The role of annealing treatment on the evolution of both types of precipitates will be presented and the influence of the precipitate distribution, size and morphology on the mechanical response will be discussed.

[1] T. Allam, K.G. Pradeep, P. Kohnen, A. Marshal, J.H. Schleifenbaum C. Haase, Additive Manufacturing 36101561 (2020)

Nano-scale characterization of Metals and Alloys

P47

ATOM PROBE STUDY OF BI DISTRIBUTION AND ITS EFFECT ON GOSS TEXTURE OF GRAIN-ORIENTED SILICON STEEL WITH TRACE BI ADDITION

Yinxing Wu¹, Jian Gong², Xianhui Wang², Qian Gao², Gang Sha¹

¹*Nanjing University of Science and Technology, China*

²*Shougang Zhixin Qian'an Electromagnetic Material Co., China*

Grain-oriented (GO) silicon steel with 3 wt.%Si is able to exhibit high permeability and low core loss, if its sharp Goss ($\{110\}\langle 001\rangle$) texture can be secured with abnormal grain growth under a recrystallization treatment [1-2]. Trace element addition has a significant effect on Goss texture formation. To date, there is lack of information about precise distribution of the trace element, and hence, detailed mechanism of the trace element addition-related texture formation remains to be explored. Herein, a combination of cutting-edge atom probe tomography (APT), scanning electron microscope (SEM), electron back-scattered diffraction (EBSD) and focused ion beam (FIB) has employed to systematically study the distribution of Bi in GO silicon steels and to understand the effect of Bi addition on the abnormal growth behavior of Goss texture under different treatment conditions. Bi is not detected at grain boundaries, but is detected in grain-growth inhibitors with local enrichment (Fig.1). Importantly, the Bi-contained silicon steel has sharper Goss texture with significantly lower area fraction of residual island crystals than GO silicon steel without Bi addition, under the same heat treatment temperature. The distribution of Bi is important for regulating the microstructure evolution and properties of GO silicon steel. Potential mechanisms for Bi-addition-enhanced Goss texture formation in GO silicon steel during secondary recrystallization will be discussed.

Nano-scale characterization of Metals and Alloys

P48

ANALYSIS OF THE EFFECT OF ELEMENT Y ON THE ANTIOXIDATION BEHAVIOR OF CRYO-MILLED OXIDE-DISPERSION-STRENGTHENED FERRITIC STEEL

Won-Sang Shin¹, Sung-Il Baik², David N. Seidman², Kee-Ahn Lee¹, Changkyoo Park³, Yoon-Jun Kim¹

¹*Inha University, South Korea*

²*Northwestern University Center for Atom-Probe Tomography, USA*

³*Seoul National University of Science and technology, South Korea*

Oxide-dispersion-strengthened (ODS) steel exhibits excellent creep and oxidation resistance, and therefore is used in severe environments such as nuclear reactors. Its nanometer-sized oxide features and the use of a combination of appropriate factors and parameters, such as milling methods, temperature, and time, for its processing enable the steel to withstand high-temperature degradation. Y-Ti-O nanometer-sized particles (NPs) with a diameter of less than 5 nm play a key role in the properties of these materials. Therefore, the oxidation resistance characteristics of ODS steel manufactured by ball milling at two temperatures were analyzed through various techniques via Atom-Probe Tomography (APT). ODS Steel were manufactured by ball milling with Y_2O_3 powder at two temperatures, room temperature and cryotemperature (≈ -150 °C). Both samples were exposed to air for 6000 h at 800 °C, and after long-term exposure both samples showed subsurface degradation occurred concurrently such as internal precipitation, phase formation, and phase dissolution. The resulting oxide scaling kinetics were more sluggish for cryotemperature compared to room temperature condition. Construction of isoconcentration surfaces of the top most oxide layer in cryotemperature sample reveals Y layer formed at the boundary between substrate and oxide layer suggesting Y atoms reacted first with oxygen to form Y_2O_3 . On the basis of the reduction of the interfacial energies calculated by using the Gibbsian interfacial excess and the partial radial distribution functions of major alloying elements, Y atoms in room temperature condition were found to show a strong tendency to form Y-Ti-O nanoparticles, while N atoms did not form NPs on the surface. On the other hand, N atoms at cryotemperature condition participated in the formation of Y-Ti-O(N) NPs, and was prone to transformed into TiN precipitates to reduce the interfacial energy as the oxidation process progressed. The transformation from Y-Ti-O(N) to TiN precipitate drove Y atoms to be released from NPs and segregated at the matrix/oxide interface. Segregated Y atoms effectively prevented further oxidation of the matrix, and thus oxide scaling kinetics became slower than solutioned in Y-Ti-O NPs.

Nano-scale characterization of Metals and Alloys

P49

NANO-SCALE INVESTIGATION OF FRACTAL ABNORMAL GRAINS AND GRAIN BOUNDARIES IN PD-AU USING ATOM PROBE TOMOGRAPHY

Johannes Wild¹, Torben Boll¹, Fabian Andorfer², Jules M. Dake², Dorothée Vinga Szabó¹, Stefan Wagner¹, Carl E. Krill², Astrid Pundt¹

¹Karlsruhe Institute of Technology, Germany

²Ulm University, Germany

Grain growth is a spontaneous process driven by the reduction of excess energy stored in the interfaces of a polycrystalline material. The microstructure of a specimen undergoing grain growth is characterized by smooth grain boundaries and a unimodal grain size distribution. However, in nanocrystalline (NC) Pd-Au prepared by inert gas condensation, we observe fractal abnormal grain growth during heat treatment [1]. The resulting microstructure exhibits a bimodal grain size distribution with micrometer-sized grains (FG) embedded in a matrix of NC grains (NG). The interfaces at the boundary between these two regions are highly irregular and resemble those of fractals. To elucidate this unusual manifestation of grain growth, we used a FIB lift-out method to extract sharp tip samples from FG and NG bulk regions, as well as from the FG-FG and FG-NG interface regions and analyzed them with a LEAP 4000X HR atom probe. The NG region exhibited an O content that was multiple times higher in comparison with the FG region. Cluster analysis confirmed the presence of a multitude of O-rich clusters in the NG, but not in the FG region. Furthermore, the reconstructions of the boundary regions revealed an unexpected diversity of host and contamination element distributions at the FG-FG and FG-NG interfaces. Some of these boundaries presented primarily with Au-depletion and Pd-enrichment, while others were defined by the segregation of either C and/or Mo to the interface.

[1] Braun, C., Dake, J.M., Krill III, C.E. et al. Abnormal grain growth mediated by fractal boundary migration at the nanoscale. *Sci Rep* 8, 1592 (2018).

Nano-scale characterization of Metals and Alloys

P50

THE CONTRIBUTION OF APT TO COMPUTATIONAL MATERIALS DESIGN FRAMEWORK FOR PRECIPITATION-ENGINEERED HIGH-PERFORMANCE MARTENSITIC STEELS

Sonia Guehairia¹

¹KTH Royal Institute of Technology, Sweden

Martensite is a key constituent for developing high-performance steels with excellent combination of strength and toughness. Fresh martensite, formed through fast cooling of the high temperature phase austenite, is normally brittle, and a tempering treatment is usually needed to induce sufficient ductility and toughness. However, the strength is decreased by tempering unless the steel is alloyed with precipitation-forming elements, to stimulate clusters and nano-particles during the tempering process. In order to develop precipitation-strengthened high-performance martensitic steels, the project has been working on the development of a computational materials design framework [1-6], instead of using the conventional trial-and-error approach. The aim is to cut significant time and resources in the steel development process. Advanced characterization tools are important for this development, as they are supporting quantitative data for database development, input for the setup of modelling, and the calibration/validation of modelling. Among them, Atom Probe Tomography (APT), with the capability of 3D atom-by-atom mapping, allows us to quantitatively characterize the precipitation kinetics, *i.e.* the evolution of chemistry, size, volume fraction, number density of precipitates with time during the tempering treatment. These quantitative data work as important inputs for developing both precipitation kinetics models and mechanical property models. In this talk, we will show how the APT, together with other characterization tools, have been supporting the development of precipitation kinetics modelling of the nucleation, growth and coarsening of Cu precipitate [3] and the development of mechanical property modelling of the stress-strain curves [5] of a maraging stainless steel 15-5 PH.

[1] T. Zhou, J. Faleskog, R.P. Babu, J. Odqvist, H. Yu, P. Hedström. *Mater. Sci. Eng.*, 745 (2019) 420-428

[2] T. Zhou, R.P. Babu, H. Yu, P. Hedström. *Mater. Des.*, 143 (2018) 141-149

[3] Z. Sheng, M.B. Rolland, T. Zhou, J. Odqvist, P. Hedström. *J. Mater. Sci.*, 56 (2021) 2650-2671

[4] T. Zhou, B. Neding, S. Lin, J.C. Tseng, P. Hedström. *Scr. Mater.*, 22 (2021) 114007

[5] P. Croné, T. Zhou, P. Hedström, J. Odqvist, P. Gudmundson, J. Faleskog. *Mater. Des.*, 215 (2022) 110463

[6] T. Zhou, G. Spartacus, A. Dahalström, R.P. Babu, A. Davydok, P. Hedström, *Scr. Mater.*, 232 (2023) 115496

Nano-scale characterization of Metals and Alloys

P51

A NOVEL HEAT-TREATMENT SOLUTION ASSISTED BY ATOM PROBE TOMOGRAPHY TO MITIGATE THE LOW-TEMPERATURE EMBRITTLEMENT IN A SUPER DUPLEX STAINLESS STEEL

Alexander Dahlström¹, Wangzhong Mu¹, Libang Lai^{1,2}, Joakim Odqvist¹, Peter Hedström¹

¹*KTH Royal Institute of Technology, Sweden*

²*Helmholtz-Zentrum Dresden-Rossendorf, Germany*

Duplex stainless steel (DSS), 2507 (UNS S32750), is a high-alloy grade super-duplex stainless steel. Due to very good chloride corrosion resistance and very high mechanical strength, this steel grade is particularly suited for use in aggressive environments such as warm chlorinated seawater and acidic chloride-containing media. For instance, it is widely used in offshore oil and gas exploration and production, in petrochemical and chemical processing heat exchangers. Besides, DSS2507 is also suitable for hydraulic and instrumentation applications in tropical marine environments. It is well-known that this alloy suffers from intermediate temperature embrittlement, which is a service lifetime-limiting phenomenon caused by a spontaneous de-mixing of Fe and Cr in the ferrite (BCC) phase. The demixing has a detrimental effect on mechanical properties resulting in severe loss of toughness. Hence, these alloys have a restricted upper service temperature of around 250°C limiting the efficiency of their applications in boilers, heat exchangers, etc. However, it is known that the initial structure of duplex stainless steel is key to predicting the early stages and temporal evolution of the de-mixing process. Thus, thermodynamic calculations are used to design a novel solution treatment and to avoid the detrimental phases during intermediate tempering. This study aims to optimize the solution treatment by introducing short-range order characterized by atom probe tomography to inhibit the onset of phase separation and prolong the service life of these alloys.

Nano-scale characterization of Metals and Alloys

P52

COUNTERING HYDROGEN EMBRITTLEMENT BY GRAIN BOUNDARY ENGINEERING: B-DOPING

Mohamed Naquib Elkot¹, Aparna Saksena¹, Binhan Sun², Dirk Ponge¹, Dierk Raabe¹

¹*Max-Planck-Institut für Eisenforschung GmbH, Germany*

²*East China University of Science and Technology, China*

The severity of hydrogen embrittlement is known to increase with the tensile strength of steels. This represents a hurdle in the processing and application of high strength steels. Hydrogen embrittlement poses the threat of intergranular fracture as a result of hydrogen-enhanced decohesion in many austenitic steels. In this work, we employ APT to investigate the solute behavior at grain boundaries of an austenitic high manganese lightweight steel alloy. We engineer the solute segregation at grain boundaries by doping the material with B. Finally, we explain the improved hydrogen embrittlement resistivity in terms of the B interaction with other solutes at the GB as observed by APT.

Nano-scale characterization of Metals and Alloys

P53

A GLIMPSE INTO THE SOLUTE DECORATION STATES AT INTERFACES IN MEDIUM-MN STEEL

Faisal Waqar Syed¹, Binhan Sun¹, Dirk Ponge¹, Dierk Raabe¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

Controlling the solute decoration state (such as dislocation and facets) at grain and phase boundaries is a commonly used technique for segregation engineering, which allows for the customization of mechanical performance in lean and inexpensive alloy compositions. Medium-Mn steels are a noteworthy group of alloys that have a complex microstructure with numerous interfaces, which significantly affects their microstructural evolution during thermomechanical treatment. In this study, Atom Probe Tomography (APT) was utilized to investigate Fe-10Mn-3Al-0.2C wt% and Fe-8Mn wt% alloys that underwent low temperature annealing for various durations and magnetic field. The mechanisms governing segregation at phase boundaries, dislocations, facets, and their crystallographic origins were examined using Transmission Electron Microscopy (TEM). Additionally, the study will explore the impact of the magnetic field on grain boundary segregation, solute drag, spinodal fluctuations, and phase evolution.

Nano-scale characterization of Metals and Alloys

P54

PRECIPITATE EVOLUTION IN AN IRON-BASED SUPERALLOY

Anna Sophie Jelinek¹, Oliver Renk¹, Lorena Juarez-Perez², Ronald Schnitzer¹

¹Montanuniversität Leoben

Iron and nickel-based superalloys are widely used for high performance applications, where high strength in combination with excellent corrosion resistance is required. This applies for aerospace engine applications as well as for the use in oil and gas industry. The main strengthening mechanism is precipitation hardening through ordered ($\text{Ni}_3(\text{Al}, \text{Ti})$) and ($\text{Ni}_3(\text{Nb})$). Besides volume fraction also morphology and chemistry of these precipitates highly influence the alloys' properties. In this work we characterize an over- and under-aged condition of an iron-based superalloy by atom probe tomography to investigate the influence of the heat treatment on the precipitate characteristics. To evaluate precipitate shape and chemistry, a correlative approach combining atom probe tomography and (scanning) transmission electron microscopy is employed. Electron microscopy is further used to analyse the atomic structure of the precipitate-matrix interface. These atomic scale investigations will be correlated to the mechanical properties and associated changes of the microstructural features caused by the applied heat treatments.

Nano-scale characterization of Metals and Alloys

P55

ANALYSIS OF FERROUS ALLOYS USING THE INVIZO 6000 ATOM PROBE MICROSCOPE

Levi Teqq¹, Ingrid E. McCarroll², Patricia Jovičević-Klug², Matic Jovičević-Klug², Hung-Wei Yen³, Julie M. Cairney¹

¹*The University of Sydney*

²*Max-Planck-Institut für Eisenforschung GmbH, Germany*

³*National Taiwan University, Taiwan*

The Cameca Invizo 6000 atom probe microscope has a laser system and ion optics which differ significantly from their earlier model local electrode atom probes (LEAPs). The result is a wider angular field-of-view, greater sample yield, and slightly improved mass resolving power compared to the LEAP4000X Si or LEAP3000 Si in our laboratories. Here we use the Invizo 6000 to study the microstructure of two ferrous alloys: a dual-precipitating low-carbon martensitic steel [1] and a martensitic stainless steel [2]. Microstructural features observed included martensite and retained austenite grains, several grain boundaries, metallic precipitates, and both nanometre and micrometre-scale carbides. Particularly for the martensitic stainless steel, the wide field-of-view and high sample yield allowed for chemical analysis of nanometre-scale carbides with stronger statistical significance than would be possible using a LEAP. Geometric distortions at the reconstruction periphery are present despite calibration of the dataset using crystallographic poles and lattice spacing. The Invizo 6000 appears well-suited to studies of ferrous alloys, particularly when searching for sparse precipitates or microstructural features with known geometry.

[1] I. E. McCarroll et al. *Scripta Materialia* 221, 114934 (2022).

[2] P. Jovičević-Klug et al. *Materials Science and Engineering: A* 873, 145033 (2023).

Nano-scale characterization of Metals and Alloys

P56

INVESTIGATING QUANTIFICATION BIAS IN NITRIDES

Sylvain Nulli¹

¹Université de Rouen Normandie, France

Nitrogen is an element that is commonly used in the industry to create protective coating on engineering pieces which endure extensive surface stress through their uses. Those coating, also referred as nitride, can greatly enhance the surface properties of the metal provided the right mixture and process are met. The Atom Probe Tomography (APT) is a chemical element measurement tool allowing to see individual atoms, clusters and boundaries giving us the ability to visualize in 3D materials at the nanoscale. While APT has proven to give incredible result for metals, some materials such as nitride are not as straightforward to analyze due to many biases affecting the data. Commonly referred as the “missing nitrogen”, nitride analyzes with APT instrument systematically reveal erroneous nitrogen content.[1] All APT experiments suffer from detection issues from multi-hit which are related to more than one atom evaporating on the same pulse. Moreover, multiples ions landing at the same time and place on the detector generally induce a single signal. This effect is known as pile-up effect. Nitride are especially affected by that phenomenon and can have up to 40% of their impact count as multi-hit.[2] Additionally, due to the way nitrogen evaporates, it tends to group up and leave as small clusters of single and molecules. Molecular ions create additional peak and can overlap with other elements. A common loss is between m/z 28 and 44, both of which reach an identical mass over charge ratio leading to an overlap of their signal. Both those issues lead to a significant portion of those nitrogen ions to be lost making analysis hazardous.[3] Nitride properties being extremely dependent on the quantity of nitrogen implemented in the matrix to form the thin coating layer, a better mean to accurately and reliably quantify nitride content is needed.

[1] W.Sha etall. Some aspects of atom-probe analysis of FeC and FeN systems, Surface Science, Volume266, 1992

[2] Z.Peng etall, On the detection of multiple events in atom probe tomography, Ultramicroscopy, Volume189, 2018

[3] J.Takahashi etall, Quantitative Analysis of Nitrogen by Atom Probe Tomography Using Stoichiometric -Fe4N Consisting of 15N Isotope, Microscopy and Microanalysis, 2022

Nano-scale characterization of Metals and Alloys

P57

SOLUTE CLUSTERING CHARACTERISTICS IN A HIGH-STRENGTH AL-MG-SI-CU ALLOY

David Tweddle¹, Jonathan A. Johnson¹, Monica Kapoor², Ilias Bickmukhametov¹, Sean Mileski², John E. Carsley², Gregory B. Thompson¹

¹University of Alabama, USA

²Novelis Global Research & Technology Center

The early stage of atomic clustering is critically important in identifying how solutes partition toward the precipitation pathways that form strengthening phases. Using atom probe tomography (APT), a difference in clustering behaviour was characterized in natural and artificially aged states that revealed the formation of Si-rich clusters that did not readily grow into Guinier-Preston zones and β'' precipitates, the primary strengthening phases for an Al-Mg-Si alloy. However, if the alloy was pre-aged followed immediately by artificial aging, a high density of β'' precipitates formed, and yielded a higher strength. Cu was found in all strengthening particles approximately 1 nm or larger and is deemed essential for the faster precipitation kinetics to enable growth of β'' precipitates. In addition, pre-aging followed by an extended secondary natural aging treatment for 30 days and subsequent artificial aging revealed a modestly lower yield strength than the direct pre-age with immediate artificial aging. This drop in strength is linked to the formation of less-beneficial Si-rich clusters that evolved over the extended natural aged period.

Nano-scale characterization of Metals and Alloys

P58

WHERE'S THE XENON? DIRECT EVIDENCE OF PLASMA FOCUS ION BEAM INFLUENCE ON ATOM PROBE SPECIMEN PREPARATION

David Tweddle¹, Jonathan A. Johnson¹, Monica Kapoor², Ilias Bikhmetov¹, Sean Mileski², John E. Carsley², Gregory B. Thompson¹

¹University of Alabama, USA

²Novelis Global Research & Technology Center

It is well reported that gallium ion focused ion beam (FIB) milling results in the implantation of the ion species into the specimen during annular milling of atom probe needle shapes that is required for field evaporation. This is particularly concerning for aluminum alloys, where gallium partitions to the grain boundaries and causes grain boundary embrittlement. With the advent of xenon ions for plasma FIB (PFIB) milling, these concerns can now be mitigated. Furthermore, xenon, as a gaseous atom, can diffuse out of the specimen prior to field evaporation in the atom probe resulting in it not being detected; this can then lead to the conclusion that xenon PFIBs yield less artifacts during specimen preparation. In this study, an Al-Mg-Si-Cu alloy has been prepared by electro-polishing (control), gallium FIB annular milling, and xenon PFIB annular milling to assess potential specimen preparation artifacts within the alloy by specifically quantifying early stages of clustering, which is sensitive to available vacancy concentrations. While no xenon was observed in the PFIB specimen, a significantly higher cluster density was observed in the PFIB prepared specimens ($\approx 25.0 \times 10^{23} \text{ m}^{-3}$) as compared to electropolished specimens ($\approx 3.2 \times 10^{23} \text{ m}^{-3}$) and gallium ion FIB samples ($\approx 5.6 \times 10^{23} \text{ m}^{-3}$). Hence, the absence of the ion milling species does not necessarily mean an absence of specimen preparation defects. Specifically, the FIB-prepared samples had more Si-rich clusters as compared to electropolished samples, which is indicative of ion impact damage increasing in the vacancy-stabilization via solute clustering.

Nano-scale characterization of Metals and Alloys

P59

MICROSTRUCTURAL IMPACTS ON OXIDATION OF MULTI-PRINCIPAL ELEMENT ALLOYS

Michael J. Pavel¹, Mark L. Weaver¹

¹*University of Alabama, USA*

The high temperature oxidation of multi-principal element alloys (MPEAs) has been predominantly performed on as-cast or homogenized microstructures which have been shown to differ in their behavior from those which have been thermally and mechanically processed. Processing conditions play an enormous role in the useful production of any material and will undoubtedly be employed if MPEA-type materials make their way into commercial engineering projects. This study explored the impacts of specific thermal processing steps on precipitate chemistry, morphology, and subsequent oxidation behavior at 1000°C during non-isothermal and cyclic testing using correlative atom probe tomography and analytical electron microscopy techniques. Thermogravimetric analysis and traditional characterization revealed that smaller precipitates had an increased capacity to coalesce and form protective sub-surface oxide layers which mitigated total mass gain. The smaller precipitate containing samples exhibited a decrease in the thickness of primary oxide scales and parabolic growth rate. The mechanism for this behavior is believed to stem from the increased growth rate of initial alumina nuclei and decreased inter-precipitate spacing which results in faster lateral diffusion and agglomeration.

Nano-scale characterization of Metals and Alloys

P60

QUANTITATIVE ANALYSIS OF VACANCY DISTRIBUTION IN METALS: EXPERIMENTS AND SIMULATIONS

Michal Dagan¹, Baptiste Gault², Julien Guérolé³, George D.W. Smith¹, Paul A.J. Bagot¹, Michael P. Moody¹

¹*University of Oxford, United Kingdom*

²*Max-Planck-Institut für Eisenforschung GmbH, Germany*

³*Université de Lorraine, France*

Precise characterization of the arrangement of individual vacancies within solids remains a frontier in high-resolution microscopy. Vacancies underpin solid-state diffusion in all crystalline materials, however, understanding their role within components for nuclear reactors is critically important. Elevated populations, induced by irradiation, can promote microstructural evolution, and hence deterioration in properties. Tungsten is a leading candidate for plasma-facing components in fusion reactors. Here, we deploy field ion microscopy, developing image processing to automatically extract 3D locations of individual vacancies within ion-irradiated tungsten. We demonstrate quantitative distribution analysis, including discrimination of individual vacancies and clusters. We compare experimental results with SRIM predictions and complementary molecular dynamics simulations, showing qualitative agreement in vacancy depth profile and cluster distribution. Results highlight a path to jointly advance characterization and computation.

Nano-scale characterization of Metals and Alloys

P61

INFLUENCE OF DEFORMATION AND PRE-AGEING ON NANOSTRUCTURAL EVOLUTION IN HIGH-STRENGTH ALUMINIUM 6XXX

Anthony Akinwale¹, James Famelton², J Carla Barbatti², Paul Bagot¹, Michael Moody¹

¹*University of Oxford, United Kingdom*

²*Brunel University London, United Kingdom*

Reducing the weight of automotive vehicles is highly beneficial as it enhances fuel efficiency and decreases emissions. Aluminium is an attractive option for achieving this goal due to its lower density in comparison to steel but on its own, it does not provide the sufficient strength for this entire application. Currently, most car bodies are made from steel but Al-Mg-Si-(Cu) alloys, known as Al-6XXX series alloys, are being investigated as potential alternatives for they provide a good combination of high strength with an ease of processing. The strength in these alloys arise from features at the nanoscale within the structure of the alloy. Atomic-scale characterisation of these features, correlated to processing conditions, will enable us to optimise the resultant microstructure and thus mechanical properties. Notably, it is understood that the distribution of very small solute clusters that can arise during the pre-ageing of 6XXX alloys at room temperature, influence subsequent microstructural evolution and are ultimately detrimental to mechanical properties [1-3]. Atom probe tomography (APT) is unique in its ability to observe and characterise the solute clusters that form during natural ageing and pre-ageing of these alloys, providing both spatial and chemical information at the very early stages of precipitation. An aluminium 6XXX alloy has been pre-aged, deformed, and final aged and APT has been used for microstructural characterisation across multiple stages of this process. The results have also been correlated with TEM data on the same alloy. The new insights provide a better understanding of the full precipitation sequence in this alloy. A study of the influence of natural ageing as well as the impact of deformation on composition, size distribution, number density and morphology of clusters and precipitates will be presented. The analysis method and approach utilised to extract this information from the APT data will also be discussed.

[1] Edwards, G., Stiller, K., Dunlop, G., & Couper, M. (1998). The precipitation sequence in Al–Mg–Si alloys. *Acta Materialia*, 46(11), 3893-3904.

[2] D.W. Pashley, J.W. Rhodes, A. Sendorek *J. Inst. Metals*, 94 (1966), p. 41

[3] Pashley, D. W., Jacobs, M. H. and Vietz, J. T., *Phil. Mag.*, 1967, 51

Nano-scale characterization of Metals and Alloys

P62

ANALYSIS OF MULTIPLE-ION EVENTS IN ATOM PROBE TOMOGRAPHY STUDIES OF MONBTI AND HFNBTATIZR MULTIPLE PRINCIPAL ELEMENTS ALLOYS

Patrick G Callahan¹, Keith E Knipling¹, David Beaudry²

¹US Naval Research Laboratory, USA

²Johns Hopkins University, USA

Multiple principal element alloys (MPEAs) are a class of alloys containing five or more primary elements, typically in nearly equiatomic proportions. This new class of alloys has significantly expanded the number of accessible alloy composition spaces and with that, potentially attainable properties. This has led to an increase in interest in refractory MPEAs for use as structural materials in extreme environments and a focus on better understanding the physical mechanisms controlling mechanical behavior in these alloys. Atom-probe tomography (APT) possesses a unique combination of high chemical sensitivity and spatial resolution, making it well-suited for uncovering physical phenomena such as atomic clustering, ordering, and the level of interstitial content that control the mechanical properties in these alloys. Here we show the effects of APT analysis parameters such as pulse energy, pulse frequency, and specimen temperature on the analysis yield, data quality, and the formation of complex ions in the two refractory MPEAs: MoNbTi and HfNbTaTiZr. By studying the MoNbTi APT mass spectra for single-hits and multiple-ion events, or multi-hits, significantly different behavior is observed for Ti than for Nb and Mo. Ti is primarily detected in single hit events, while Mo and Nb evaporate as single- and multi-hit events in similar proportions. These multi-hit events have been studied using ion correlation histograms, indicating that as much as 40% of the detected Mo and Nb arrive in multi-hit events, and that molecules such as NbN and NbO are detected almost exclusively in multi-hit events. There is also significant molecular dissociation of complex ions containing H and Mo or Nb. In the case of HfNbTaTiZr, considering the multi-hit mass spectra is absolutely necessary to unambiguously differentiate between Hf, Zr, and Ta in ions detected near 90 Da and 180 Da. Examples will be shown to illustrate these issues.

Advances in Data Reconstruction & Data Analysis, Machine Learning Approaches, Simulation Tools, Open Software

P63

ADAPTIVE RECONSTRUCTION: A METHOD TO ADAPT THE RECONSTRUCTION PROJECTION TO A REALISTIC SPECIMEN SHAPE

David A. Reinhard¹, Brian P. Geiser¹, Joseph Bunton¹, David J. Larson¹

¹CAMECA Instruments, Inc.

Standard atom probe tomography (APT) reconstructions rely on an assumption of a spherical apex shape typically in conjunction with an analytical projection to recreate spatial positions for detected ion events. [1, 2] This is a clear limitation because it is known that APT specimens generally do not have a spherical apex. [3] To enable reconstructions of APT data collected from aspherical specimen apex shapes we have developed a reconstruction method that can take in any specimen shape, create arrays of point charges around it, and map out the trajectories of ions from these point charges to the detector through an electrostatic model of the atom probe. [4,5] Here we present a series of comparisons from simulated datasets between the classical APT reconstruction based on a projection from a spherical apex and the new adaptive reconstruction method based on projecting ions from any apex shape through an electrostatic model.

[1] P. Bas, et al., Appl. Surf. Sci. 87/88 (1995) 298.

[2] De Geuser, F. and B. Gault, Microscopy and Microanalysis 23 (2017), p. 238. doi: 10.1017/S1431927620022217

[3] D. J. Larson et al., Microscopy and Microanalysis 18(5) (2012), p. 953

[4] B. Geiser et al., Microscopy and Microanalysis 26 (2020), p. 2622. doi: 10.1017/S1431927619002411 [5] B. Geiser et al., Microscopy and Microanalysis 27 (2021), p. 2482. doi: 10.1017/S1431927621008850

Advances in Data Reconstruction & Data Analysis, Machine Learning Approaches, Simulation Tools, Open Software

P64

ADAPTIVE RECONSTRUCTION: A METHOD TO ADAPT THE RECONSTRUCTION PROJECTION TO A REALISTIC SPECIMEN SHAPE

David A. Reinhard¹, James E. Payne¹, Benjamin Klaes², Eric Strennen¹, Brian P. Geiser¹, Gerald Da Costa²

¹*CAMECA Instruments, Inc.*

²*University of Rouen Normandie, France*

As the user base and number of applications continue to grow in atom probe tomography (APT), ideas for new analysis tools continue to grow as well. These innovative techniques are typically not immediately available in established analysis software platforms. The result is a growing need for a platform to easily develop and share software to reduce duplication in efforts [1,2]. CAMECA® has made substantial enhancements to the extension infrastructure in the AP Suite™ 6.3 software to support developing, distributing, and running custom analyses. This includes tools, documentation, and examples to support extension development. Public code libraries enable developing extensions without requiring an installed instance of AP Suite. Available data and functionality include access to reconstructed position, mass, and ion type information. Additional data can be written to and read from the *.APT data file [3]. Custom user interfaces for an analysis can be designed, and several native data visualization elements are supported to provide seamless visual integration with AP Suite, including multidimensional charts, histograms, and tables. If a native chart is not available, knowledge of Windows Presentation Foundation development can aid in creating new forms of display. A quick start document guides the process of creating a new extension utilizing a publicly available template. By utilizing these tools, developers can focus entirely on implementation of their analysis and more easily share it with other researchers via the most widely used APT analysis software. Updated documentation, guides, and examples are available on GitHub [4]. Users can add or remove extensions directly from AP Suite; no additional tools are required. These extensions can be kept locally for private use or stored in a public repository to enable access for any user of AP Suite with an internet connection. As an example of this workflow, CAMECA publishes several specialized analyses that can be installed [4].

[1] M. Kuhbach, P. Bajaj et al., *npj Comput Mater.* 7 (2021).

[2] I Ghamarian & E. A. Marquis, *Ultramicroscopy.* 200 (2019) p.28.

[3] CAMECA Instruments Inc., *AP Suite User Guide*, (2022) p.B-2.

[4] *Cameca Instruments, Inc. – Atom Probe Tomography*, <https://github.com/CamecaAPT> (accessed May 2, 2023).

Advances in Data Reconstruction & Data Analysis, Machine Learning Approaches, Simulation Tools, Open Software

P65

COUPLING SOFTWARE TOOLS FOR REPRODUCIBLE DATA ANALYSIS WORKFLOWS IN ATOM PROBE

Markus Kühbach¹, Sarath Menon², Alaukik Saxena², Mariano Forti³, Aparna Saksena², Christoph Freysoldt², Sandor Brockhauser¹, Tilmann Hicke⁴, Thomas Hammerschmidt³

¹Humboldt-Universität zu Berlin, Germany

²Max-Planck-Institut für Eisenforschung GmbH, Germany

³Ruhr-Universität Bochum, Germany

⁴Bundesanstalt für Materialforschung und -prüfung (BAM), Germany

Atom probe tomography and related field-ion microscopy became mature nanoscale structure and composition characterization methods^[1, 2]: A technique to gain a more detailed understanding of microstructure-materials-properties relations and a rich collection of distributed point-cloud datasets for developing new and improving existent data-analysis techniques. There are several possibilities for improving the current state-of-the-art in terms of how software for analyzing atom probe data gets developed, how such software is being used, and how we can support scientists who would like to share datasets and results more frequently between laboratories^[3, 4]. The German Federal Government has set up a funding scheme^[5] to build an interdisciplinary network of research consortia which develop software tools and community standards for improving the management and utilization of research data. These efforts are guided by the FAIR^[6, 7] principles. In our contribution, we deliver an update on the progress within a cooperation between the NFDI-MatWerk^[8] and FAIRmat^[9] consortia on coupling different tools for analyzing atom probe data and showing their interoperability^[8-11]. We will focus on an example of characterizing composition and structural features of interfaces in an engineering alloy.

[1] B. Gault et al., *Nat. Rev. Methods Primers* 1 (2021), <https://doi.org/10.1038/s43586-021-00054-x>

[2] S. Katnagallu et al., *New J. Phys.* 21 (2019), <https://doi.org/10.1088/1367-2630/ab5cc4>

[3] E. Marquis et al., Proc. 18th Int. Conf. on Environmental Degradation of Materials in Nuclear Power Systems (2017), https://link.springer.com/chapter/10.1007/978-3-030-04639-2_141

[4] M. Kühbach et al., *Microsc. Microanal.* 28(4) (2021), <https://doi.org/10.1017/S1431927621012241>

[5] <https://www.eosc-pillar.eu/news/nfdi-german-national-research-data-infrastructure-york-sure-vetter>

[6] M. D. Wilkinson et al., *Scientific Data* 3 (2016), <https://doi.org/10.1038/sdata.2016.18>

[7] M. Barker et al., *Scientific Data* 9 (2022), <https://doi.org/10.1038/s41597-022-01710-x>

[8] C. Eberl et al., v1, Jul 8th, 2021, <https://zenodo.org/record/5082837#.YubgaxxBy38>, <https://nfdi-matwerk.de/infrastructure-use-cases/iuc09-infrastructure-interfaces-with-condensed-matter-physics-collaboration-with-fairmat>

[9] M. Scheffler et al., *Nature*, 604 (2022), <https://doi.org/10.1038/s41586-022-04501-x>, <https://www.fairmat-nfdi.eu/fairmat/>

[10] Jan Janssen et al., *Comput. Mater. Sci.* 163 (2019), <https://doi.org/10.1016/j.commatsci.2018.07.043>

[11] <https://fairmat-nfdi.github.io/nexus-fairmat-proposal> All webpages were accessed May, 8th 2023 the last time. [4] *Cameca Instruments, Inc. – Atom Probe Tomography*, <https://github.com/CamecaAPT> (accessed May 2, 2023).

Advances in Data Reconstruction & Data Analysis, Machine Learning Approaches, Simulation Tools, Open Software

P66

ESTABLISHING THE ELEMENTAL HOMOGENEITY WITHIN HIGHLY BORON DOPED (~4 AT.%) SiGe LAYER USING ATOM PROBE TOMOGRAPHY

Jhao-Rong Lin^{1,2}, Richard J. H. Morris¹, Jeroen E. Scheerder¹, Andriy Hikavyi¹, Clement Porret¹, André Vantomme², Claudia Fleischmann^{1,2}

¹IMEC, Belgium

²KU Leuven, Belgium

For Si and SiGe, boron remains the p-type dopant of choice. Due to device scaling there is a push to enhance these doping levels to >1 at.% to reduce the source drain resistance. The boron spatial distribution and positioning within the lattice i.e., substitutional/interstitial, at these enhanced doping levels is an important parameter. The availability of accurate and dependable metrology to quantify the localized composition around different elements is therefore required. Although atom probe tomography holds the potential to meet this requirement, the accuracy will undoubtedly be influenced by the experimental conditions and reconstruction approach. In this presentation, a preliminary study regarding the homogeneity analysis capabilities has been conducted using a 43 nm thick highly boron doped (~4 at.%) SiGe layer epitaxially grown and with an intended Si:Ge site fraction of 0.7:0.3. Atom probe analysis was performed using a Cameca LEAP 5000XR in UV laser mode ($\lambda = 355$ nm). A laser pulse energy of 20 pJ yielding a Si charge state ratio (CSR) of $\text{Si}^{++}/(\text{Si}^{+}+\text{Si}^{++})\sim 0.95$ was used. For this Si CSR an accurate composition profile is expected [2]. A voltage reconstruction profile to obtain the known SiGe layer thickness was applied. An in-house Python code using the algorithm reported in reference 3 was used to assess the elemental homogeneity of our dataset. The code uses a fixed number of nearest neighbor atoms surrounding the atomic species of interest i.e., it enables the local concentration about each atom of interest to be determined. Subsequently, the resulting concentration frequency histogram for our experimental dataset was compared with a randomized version of the same dataset to assess the degree of inhomogeneity. Our initial results indicate that ~20 % of the boron atoms differ in their spatial distribution compared to that predicted by the randomized dataset, indicating significant inhomogeneity is present. The next step is to establish the accuracy, reproducibility and limitations of such an approach.

[1] G. Rengo *et al.*, *ECS Transactions*, 98 (2020) 27–36.

[2] M. Dialameh, PhD Thesis, KU Leuven, Belgium (2019).

[3] J.M. Hyde *et al.*, *Ultramicroscopy*, 109 (2009) 502–509.

Advances in Data Reconstruction & Data Analysis, Machine Learning Approaches, Simulation Tools, Open Software

P67

CHEMICALLY-SENSITIVE IMAGING OF ATOM PROBE TIPS BY A CONTACT-LESS, LENS-FREE PTYCHOGRAPHIC METHOD

Vitaly Krasnov^{1,2}, Igor Makhotkin^{1,3}, Jeroen E. Scheerder¹, Lars Loetgering⁴, Victor Soltwisch⁵, Paul van der Heide¹, Claudia Fleischmann^{1,2}

¹IMEC, Belgium

²KU Leuven, Belgium

³University of Twente, The Netherlands

⁴Friedrich-Schiller-University Jena, Germany

⁵Physikalisch-Technische Bundesanstalt, Germany

Determining the evolving shape of an atom probe tip (in real time) during analysis can be an important cornerstone for an accurate and precise 3D-reconstruction in atom probe microscopy¹. Here, we present a proof-of-concept study that evaluates for the first time the feasibility and limitation of a contact-less, lens-free imaging technique, i.e. soft x-ray ptychography for such a purpose. Ptychography is a form of coherent diffractive imaging (CDI) technique where the sample is scanned with a focused light beam. The technique allows to reconstruct both amplitude and phase distributions in the sample plane, much like holography, via customized iterative phase retrieval algorithms. Utilizing information on both absorption and phase shift in every pixel of the sample's image enables chemical resolution. The large number of collected diffraction patterns provides a tremendous level of data redundancy, which allows to achieve both high resolution and high signal-to-noise ratio. This, and additional inherent advantages of CDI, namely that it is: contact-less and non-destructive made ptychography quite popular in recent years. Leveraging the chemical sensitivity of ptychography, we were able to image, in 2D, the geometry of an atom probe needle and to visualize embedded, nano-meter sized features with a resolution down to 11 nm (at 800 eV). In this study, atom probe tips were imaged at the MAXYMUS scanning x-ray microscope at the electron storage ring BESSY II. The prospect of implementing this imaging method in the laboratory/atom probe tool with a table-top 92 eV high harmonic generation (HHG) source will be discussed, together with an outlook on how to obtain a 3-dimensional image of an atom probe tip. We will summarize the limitations and potentials of this lens-free imaging technique in view of its usefulness for advanced data reconstruction schemes.

[1] Op De Beeck, J. *et al.* The Prospect of Spatially Accurate Reconstructed Atom Probe Data Using Experimental Emitter Shapes. *Microscopy and Microanalysis* 28, 1141–1149 (2022).

Advances in Hydrogen Detection

P68

RE-EVALUATING OUR UNDERSTANDING OF MAGNESIUM OXIDATION USING ATOM PROBE TOMOGRAPHY

Tim Schwarz¹, Baptiste Gault¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

As we develop our scientific knowledge we lay out foundations for how we understand the world. As new scientific techniques are developed we obtain deeper insight into certain processes, occasionally revealing flaws in these scientific foundations. Over the past decade I have been applying the technique of atom probe tomography to the study of magnesium oxidation with rather surprising results. In this presentation I will detail the existing theories related to the early oxidation processes of magnesium alloys and outline discrepancies between these theories and results from atom probe studies of magnesium oxidation experiments.

Advances in Hydrogen Detection

P69

ROLE OF ALLOYING ELEMENTS IN THE OXIDATION AND HYDROGEN EMBRITTLEMENT SUSCEPTIBILITY OF TWINNING INDUCED PLASTICITY STEELS STUDIED BY ATOM PROBE TOMOGRAPHY

Heena Khanchandani¹, Stefan Zeiler¹, Lucas Strobel¹, Mathias Goeken¹, Peter Felfer¹, Baptiste Gault^{2,3}

¹Friedrich-Alexander University Erlangen-Nürnberg, Germany

²Max-Planck-Institut für Eisenforschung GmbH, Germany

³Imperial College London, United Kingdom

Twinning induced plasticity (TWIP) steels are promising materials for automotive applications [1]. However, they are susceptible to oxidation [2] and hydrogen embrittlement (HE) during service loading conditions [3]. We investigated the mechanisms underpinning the oxidation and HE in a model Fe-27Mn-0.3C (wt.%) TWIP steel by atom probe tomography. We measured the segregation of hydrogen and oxygen at grain boundaries. We also studied the role of carbon and manganese in the oxidation and HE susceptibility which are major alloying elements in TWIP steels. This study led to the design of a novel steel which was exposed to the electrochemical hydrogen charging and its mechanical properties did not change following the hydrogen charging. The results on TWIP steel which motivated the design of novel steel concept and its design will be discussed in details during the talk.

[1] B.C. De Cooman, O. Kwon, K.G. Chin, *Mater. Sci. Technol.* 28 (2012) 513–527.

[2] D.M. Bastidas, J. Röss, J. Bosch, U. Martin, *Metals (Basel)*. 11 (2021) 1–45.

[3] M. Koyama, E. Akiyama, K. Tsuzaki, *Scr. Mater.* 66 (2012) 947–950.

Advances in Hydrogen Detection

P70

ATOM PROBE EXPERIMENTS WITH SPECIMENS UNDER PRESSURIZED HYDROGEN

Benedict Ott¹, Martina Heller¹, Mehrpad Monajem¹, Peter Felfer¹

¹Friedrich-Alexander University Erlangen-Nürnberg, Germany

Atom probe tomography is currently the only microanalytical method capable to image and quantify hydrogen at plasticity defining crystal defects. So far, a number of studies have been undertaken using electrochemical hydrogen charging [1] and gaseous hydrogen charging [2]. However, the electrochemical charging results are hard to quantify and the gaseous charging studies were conducted at low pressures (typ. < 1 bar) for safety reasons. In order to create thermodynamically defined states of hydrogen charging, in this work we present experimental setups that expand the pressure range that was accessible so far to pressures up to 50 bar using deuterium and up to 1000 bar using regular hydrogen. The ‘low pressure’ (< 50 bar) setups are simple lab scale devices, that by using very small volumes can be used standalone with no further safety infrastructure. They can easily be filled with deuterium from a bottle. The high-pressure setup is a pressure autoclave reaching 1000 bar at up to 300 °C from a compressor and is housed in a safety bunker. At current, this system has no H recirculation, thus making tracer experiments prohibitively expensive. Using these setups, we will present experiments on metallic materials, where we used our ultra-low H atom probe [3] in conjunction with our cryo transfer system [4] to analyse H-defect interactions.

[1] Y.-S. Chen *u. a.*, „Direct observation of individual hydrogen atoms at trapping sites in a ferritic steel“, *Science*, Bd. 355, Nr. 6330, S. 1196–1199, März 2017, doi: 10.1126/science.aal2418.

[2] P. Kesten, A. Pundt, G. Schmitz, M. Weisheit, H. U. Krebs, und R. Kirchheim, „H- and D distribution in metallic multilayers studied by 3-dimensional atom probe analysis and secondary ion mass spectrometry“, *J. Alloys Compd.*, Bd. 330–332, S. 225–228, Jan. 2002, doi: 10.1016/S0925-8388(01)01596-1.

[3] P. Felfer *u. a.*, „An Atom Probe with Ultra-Low Hydrogen Background“, *Microsc. Microanal.*, Bd. 28, Nr. 4, S. 1255–1263, Aug. 2022, doi: 10.1017/S1431927621013702.

[4] C. Macauley, M. Heller, A. Rausch, F. Kümmel, und P. Felfer, „A versatile cryo-transfer system, connecting cryogenic focused ion beam sample preparation to atom probe microscopy“, *PLoS ONE*, Bd. 16, Nr. 1 January, 2021, doi: 10.1371/journal.pone.0245555.

Advances in Hydrogen Detection

P71

DIRECT OBSERVATION OF HYDROGEN IN STEEL: A COMPARISON OF THE HYDROGEN TRAPPING MECHANISM OF TIC AND (TI-MO)C PRECIPITATE

Pang-Yu Liu¹, Ranming Niu¹, Patrick Bur², Yi-Sheng Chen¹, Julie Cairney¹

¹*The University of Sydney, Australia*

²*The University of New South Wales*

The presence of hydrogen in steels can lead to the reduction of ductility hence unpredictable early cracking, and this problem is known as ‘hydrogen embrittlement (HE). The introduction of hydrogen traps into alloy microstructure to restrict hydrogen diffusion towards HE-susceptible zones like microcracks has been proven to be effective against HE. Especially, nanosized transition metals (Ti, Mo, Nb, V, etc.) carbide traps can form high number density and evenly dispersed hydrogen traps in the steels. However, the mechanism of their hydrogen trapping, i.e. whether it is driven by bulk defects in the carbide or by the carbide-matrix interface, remains unknown. In this work, we adopt the atom probe tomography with cryo-transfer protocol to elucidate the hydrogen trapping mechanisms of TiC and TiMoC in ferritic steels. In our observation, two different hydrogen trapping mechanisms were observed: the deuterium may tend to reside within the TiMoC bulk, while the deuterium prefers to stay at the interface of TiC and ferrite matrix. These may be due to the existence of the carbon vacancy caused by the Mo addition, allowing the deuterium atoms to enter the bulk of the carbide.

Advances in Hydrogen Detection

P72

REACTION MICROSCOPY OF HYDROGEN AND HYDRIDES IN ATOM PROBE TOMOGRAPHY OF III-N SEMICONDUCTORS

Aissatou Diaque¹, Samba Ndiaye¹, Noelle Gogneau², Jonathan Houard¹, Lorenzo Rigutti¹

¹University of Rouen Normandie, France

We investigate the microscopic behaviour of hydrogen-containing species formed on the surface of III-N semiconductor samples by the residual hydrogen in the analysis chamber in laser-assisted atom probe tomography (APT). APT is based on the field evaporation of atoms on the surface of a nanometric tip. It has the ability to reproduce a 3D image of the distribution of atoms in a small volume of the material and to quantitatively measure the chemical composition in a small area of the volume to be analysed. We analysed $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ heterostructures containing alternate layers with a thickness of about 20 nm. The formation of H-containing species occurs at field strengths between 22 and 26 V/nm and is independent of the analysed samples. The results highlight the strong dependence of the relative abundances of the ionic species H^+ , H_2^+ , H_3^+ and hydrides on the surface field of the samples [1], estimated from the Kingham post ionisation theory using the ratio of metal ions [2]. The relative abundances of the hydrides decrease when the surface field increases due to the evolution of the tip shape or the different evaporation behaviour of the different layers. The 3D APT reconstruction makes it possible to map the evolution of the surface behaviour of these species issued by chemical reactions. This, together with the analysis of multiple, correlated evaporation events in the detector space, opens the way to a spatially resolved microscopy of single-molecule chemical reactions based on APT.

[1] L. Rigutti *et al.*, « Surface Microscopy of Atomic and Molecular Hydrogen from Field-Evaporating Semiconductors », *J. Phys. Chem. C*, vol. 125, n° 31, p. 17078-17087, août 2021, doi: 10.1021/acs.jpcc.1c04778.

[2] D. R. Kingham, « The post-ionization of field evaporated ions: A theoretical explanation of multiple charge states », *Surf. Sci.*, vol. 116, n° 2, p. 273-301, avr. 1982, doi: 10.1016/0039-6028(82)90434-4.

Advances in Hydrogen Detection

P73

MICROSCOPIC BEHAVIOUR OF HYDROGEN AND HYDRIDES IN ATOM PROBE TOMOGRAPHY OF ZIRCONIUM

Aissatou Diaque¹, Lorenzo Rigutti¹

¹University of Rouen Normandie, France

The understanding of Zirconium (Zr) and Hydrogen (H) interactions is a topic of interest in the field of materials science. Zr is known to have a strong affinity with hydrogen, which can lead to the formation of hydrides that can affect the mechanical properties (embrittlement, cracking, etc) of the material [1]. Our studies are carried out on pure Zr analysed by laser-assisted atom probe tomography. This is complex, because the hydrogen detected during the analysis could come from the analysis chamber (parasitic hydrogen) or from the material (hydrogen contained in the material). Our results show the formation of hydrogen species H^+ , H_2^+ , H_3^+ and hydrides ZrH_x^{2+} . The evolution of the relative abundances of H^+ , H_2^+ , H_3^+ depends on the surface field estimated from the Zr^{3+}/Zr^{2+} ratios, as seen in previous studies on other materials[2]. This is not the case for the hydrides which overlap with the Zr isotopic species. In this contribution we will discuss the quantification of hydrogen and zirconium hydrides and their dependence on the field.

[1] J. Bair, M. Asle Zaeem, et M. Tonks, « A review on hydride precipitation in zirconium alloys », *J. Nucl. Mater.*, vol. 466, p. 12-20, nov. 2015, doi: 10.1016/j.jnucmat.2015.07.014.

[2] L. Rigutti *et al.*, « Surface Microscopy of Atomic and Molecular Hydrogen from Field-Evaporating Semiconductors », *J. Phys. Chem. C*, vol. 125, n° 31, p. 17078-17087, août 2021, doi: 10.1021/acs.jpcc.1c04778.

Advances in Hydrogen Detection

P74

ANALYTIC MODEL FOR IN-SITU IMPLANTATION AND CHARACTERIZATION OF HYDROGEN IN APT

Jean-Baptiste Maillet¹

¹University of Rouen Normandie, France

The study of hydrogen in Atom Probe Tomography is still a relevant challenge. Its low mass, high diffusion coefficient and presence as a residual gas in vacuum chambers generate multiple complications for APT investigation. Different solutions were proposed in literature to charge our sample like ex-situ charging and cryopreparation and cryotransfer [1] or using a separate chamber connected to the APT where hydrogen charging is performed at high temperature [2]. In this paper, we follow up on the previous lecture given at APT&M22 where the alternative route in-situ implantation was validated experimentally but without an analytical model. Here we show in detail the analytical model. To remind, the basic idea is to apply an negative pulses to the APT sample with a controlled repetition rate and amplitude (-1,000V to -3,000V) under low H₂ pressure (~10⁻⁵ mbar) in APT chamber. Under the electron bombardment, some ionized gas atoms (positive ions) are accelerated towards the top of the sample (negative polarization) at an implantation energy (~200-1000eV) and at a dose (~20000-100000at). Using electrodynamic simulations, it is possible to quantitatively estimate the expected hydrogen number in the APT specimen and the depth of implantation. The analytic model considers the current emitted at the apex, the cross section between the electron and gas (H₂), the field of view of the instrument, the energies and trajectories of the particles, the negative pulse voltage applied to the specimen and the curvature radius. A comparison is then made between the model and the experimental results with their associated depth and flux of hydrogen ion implanted.

[1] Yi-Sheng Chen et al. Cryo Atom Probe: Freezing atoms in place for 3D mapping. Nano Today 37 (2021).

[2] J. Takahashi et al. Origin of hydrogen trapping site in vanadium carbide precipitation strengthening steel. Acta Materialia 153, p 193-204 (2018).

Nano-scale characterization of Nuclear and Radioactive Materials

P75

ATOM PROBE TOMOGRAPHY INVESTIGATION OF THE EVOLUTION OF ULTRAFINE-GRAINED AUSTENITIC STAINLESS STEEL UNDER NEUTRON IRRADIATION

Frederic Habiyaremye¹, Bertrand Radiguet¹, Solène Rouland¹, Xavier Sauvage¹, Benoit Tanguy², Joël Malaplate², Christophe Domain³, Remy Bonzom³, Marina Abramova⁴, Nariman Enikeev⁵, Auriane Etienne¹

¹University of Rouen Normandie, France

²Université Paris-Saclay, France

³EDF Lab Renardières, France

⁴Ufa State Aviation Technical University, Russia

⁵Saint Petersburg State University, Russia

Austenitic stainless steels (ASS) used in the internal structures of the pressurized water reactor suffer from radiation-induced precipitation and segregation. These phenomenon results from the supersaturation of point defects (vacancies and self interstitial atoms) produced by elastic collision between crystal atoms and fast neutrons. Thus, one possible approach to try to improve the irradiation resistance of ASS is to increase the density of point defects sinks such as grain boundaries. Ultrafine-grained austenitic stainless steels (UFG-ASS) have been suggested for this purpose because they have a large density of grain boundaries. This study investigates the evolution under neutron irradiation of microstructure and mechanical properties of a 316 UFG-ASS processed by equal channel angular pressing with 4 passes at 400°C and 1 pass at 500°C. Neutron irradiations of the sample were performed at CEA Saclay (France) in OSIRIS experimental reactor with different fluences of neutrons resulting in doses between 3.9 and 11.6 displacements per atom, at 350°C. Microstructural evolutions were examined with atom probe tomography. Mechanical properties were obtained from tensile tests. The results of this study were compared to the current 316L ASS behavior reported in literature to assess the potential application of this alloy for the core of Gen. II and IV nuclear reactors.

Nano-scale characterization of Nuclear and Radioactive Materials

P76

APT CHARACTERIZATION OF STAINLESS-STEEL SURFACE CONTAMINATION FROM SIMULATED REPROCESSING LIQUOR

Anne Callow¹, Thomas Carey¹, Christina Hofer², Sarah Bibby³

¹*National Nuclear Laboratory, United Kingdom*

²*Oxford University, United Kingdom*

³*Sellafield Ltd, United Kingdom*

Reprocessing spent nuclear fuel generates a large inventory of contaminated stainless-steel waste for decontamination and decommissioning. The depth and nature of contamination must be understood so that decontamination is effective and cost-efficient. Contamination-bearing cracks are present where the plant surfaces have undergone inter-granular attack (IGA), which may occur in the most corrosive aqueous conditions such as in evaporators. Atom Probe Tomography (APT) was used to characterize the contaminated oxide and metal/oxide interface on 304L and NAG18/10L stainless steel corroded in simulant reprocessing liquors. Simulant contaminated samples were generated by boiling steel coupons until IGA occurred in 8 M HNO₃ containing caesium and strontium, with 25 g L⁻¹ iron as a corrosion enhancer. A second experiment used a non-radioactive simulant liquor containing a range of elements representative of concentrated reprocessing liquor. All coupons showed evidence of IGA and grain-dropping from corrosion. APT samples were made by Focused Ion Beam lift-out from corroded plate-surface grains, after bulk coating with approximately 200 nm of Pt-Pd. The results showed very thin oxide layers of 4 nm on the sample corroded in the Cs, Sr liquor and 13 nm after corrosion in the simulant liquor, with an uneven morphology and thickness. Although thin, the contamination layer is presumed to be present along the contour of surface cracks which penetrate tens of microns into the surface. The simulant elements were found within the oxide at a reducing concentration with depth. A small amount of contamination was found within the nickel-enriched metal underneath the oxide. The steel metal mass-spectrum showed that contamination did not penetrate beyond the nickel-enriched layer. These results have implications for the decontamination strategy used for reprocessing sites. Metal dissolution during decontamination could be reduced if a thin layer can be effectively removed. Further work is required to investigate the contamination at corroded crack tips, or at deeper corrosion at microstructural features such as end-grain pits or cracks at weld sites. These reproducible simulated samples will support decontamination trials using characterization before/after decontamination. APT could be applied to real active samples with similar corrosion features and morphology, although this will generate a more complex mass spectrum.

Nano-scale characterization of Nuclear and Radioactive Materials

P77

THE EFFECTS OF TEMPERATURES AND DOSES ON GRAIN BOUNDARY SEGREGATION IN ION-IRRADIATED FE-MN-NI(-SI) STEELS

Jing Xue¹, Rong Hu², Gang Sha²

¹*Advanced materials Research Institute Yangtze Delta, China*

²*Nanjing Tech University, China*

The radiation induced segregation (RIS) generates from the migration of point defects, and strongly affects the grain boundary chemistry as well as the mechanical performance under irradiation conditions. Many parameters influence the level of RIS, such as temperature, dose, impurities and so on. This work investigates the temperature influence at 1.5 dpa and dose influence at 350 °C on RIS in Fe-Mn-Ni(-Si) steels, based on high angle grain boundaries (misorientation around 50°) via electron back-scattered diffraction (EBSD) and atom probe tomography (APT). The results revealed that the segregation levels of Mn, Ni, Si increased with the temperature and dose. In terms of C, the segregation level decreased with the temperature increasing, however the segregation level varied between 2.10 and 2.91 atom/nm² with the dose increasing. It suggests that the influence of the temperature on the grain boundary segregation is more obvious than the dose. At the same time, there is a competitive relationship between Ni and Si atoms at the grain boundary under the same irradiation conditions. In addition, the Mn-rich precipitates found in the grain boundaries were analyzed.

Nano-scale characterization of Nuclear and Radioactive Materials

P78

EFFECTS OF SI ADDITION AND TEMPERATURE ON CLUSTER EVOLUTIONS OF FE-MN-NI(-SI) STEELS UNDER FE ION IRRADIATION

Jing Xue¹, Rong Hu², Gang Sha²

¹*Advanced materials Research Institute Yangtze Delta, China*

²*Nanjing Tech University, China*

The irradiation-induced solute clusters is one of typical microstructural defects which formed under irradiation. This work addresses the cluster evolutions in Fe-Mn-Ni(-Si) steels at 350 °C with different doses (0.1 dpa, 0.6 dpa, 1.5 dpa) and at 1.5 dpa with different temperatures (room temperature, 250 °C, 350 °C), to gain the effects of Si addition and the temperature on the the nucleation, growth and chemistry evolution of MnNi(Si) clusters via atom probe tomography. At 350 °C, the clustering in the steels involves two kinetic processes, i.e. MnNi(Si) cluster chemistry evolution and size growth. The chemistry evolution by which the clusters reached a stable chemistry is accomplished prior to the fast growth of the MN clusters. The stable chemistry makes the MnNi clusters exhibit 'late-blooming' to reach larger sizes via coalescing than MNS clusters. The Si addition enhances the nucleation of MnNiSi clusters, prolongs the cluster composition evolution, and prohibits their fast growth. At 1.5 dpa, the cluster evolution under different temperatures is found to be controlled by the competition of the mobility between interstitials and vacancies. The Si addition is also found to slow down the growth of solute clusters at all temperatures due to the strong vacancy-Si interaction.

Nano-scale characterization of Nuclear and Radioactive Materials

P79

APT ANALYSIS OF CR₂O₃-COATED ZIRCALOY-4 ADVANCED TECHNOLOGY FUEL CLADDING

Alexandra Cackett¹, Calum Cunningham¹, Mattia Cabrioli², Paul Styman¹

¹*National Nuclear Laboratory, United Kingdom*

²*Politecnico di Milano*

Zirconium-based alloys have widely been used in the manufacture of fuel cladding for pressurized water reactors (PWRs) due to a favourable combination of their lower neutron absorption cross-section and good corrosion resistance. However, in a loss-of-coolant (LOCA) accident these alloys are susceptible to rapid oxidation, which leads to the production of hydrogen that has the potential to cause an explosion such as that suffered by the Fukushima Daiichi plant in 2011 [1]. Various so-called ‘Advanced Technology Fuels’ (ATFs) are being explored to tackle this problem and an initial stage in ATF research is the development of oxidation-resistant coatings that could be applied to existing Zr alloy cladding. Cr-based coatings are one promising candidate for this application [2]. As part of the IL TROVATORE (Innovative cladding maTeRials fOr adVanced AccidenT-tOlerant eneRgy systEMs) project, sections of Zircaloy-4 tube were coated with 3 μm of Cr₂O₃ using pulsed laser deposition. Using atom probe tomography (APT), this as-coated sample was analysed and a thin (~10 nm) Zr oxide layer was observed at the interface between coating and Zr alloy, which is thought to be due to the manufacturing route. A second sample of the same coated tube material was then exposed to PWR-like water in an autoclave and APT analysis repeated. Results showed a complex microstructure within the region of coating closest to the tube substrate; evidence was found of Sn having migrated from the ZrO₂ layer and becoming associated with Cr phases, as well as a reduction in Cr content as the distance from the coating surface increased.

[1] S. J. Zinkle and G. S. Was, ‘Materials challenges in nuclear energy’, *Acta Mater.*, vol. 61, no. 3, pp. 735–758, (2013)

[2] D. J. Park *et. al.*, ‘Behavior of an improved Zr fuel cladding with oxidation resistant coating under loss-of-coolant accident conditions’, *J. Nucl. Mater.*, vol. 482, pp. 75–82, (2016)

Nano-scale characterization of Nuclear and Radioactive Materials

P80

AN APT STUDY OF NEUTRON-IRRADIATION INDUCED DAMAGE IN ZIRLO ALLOYS

Wenyu Zhang¹, Paul A. J. Bagot¹, Chris R. M. Grovenor¹, Michael P. Moody¹

¹University of Oxford, United Kingdom

Zr alloys continue to serve in 3rd generation nuclear fission reactors due to their combination of outstanding corrosion and irradiation resistance and useful mechanical properties. However, to meet demands for stricter safety requirements and more efficient energy production, Zr cladding materials need to be developed that are more tolerant to the harsh in-reactor conditions. This makes better understanding how these materials degrade under neutron irradiation essential. There have been a significant number of studies characterising the redistribution of alloying elements and irradiation induced growth (IIG) in both ion- and proton-irradiated Zr alloys [1, 2]. However, many fewer studies have been carried out on neutron-irradiated materials because of the relative availability, and the cost and difficulty in handling active materials. The results have already shown that the microstructures can behave differently in comparison with their ion/proton-irradiated counterparts [3]. This inevitably casts doubts on the feasibility of utilising ion/proton irradiation conditions to simulate a real neutron-irradiated environment. In this work, ZIRLO alloys subjected to 1 to 4 neutron-irradiation cycles at the Vogtle plant (Georgia, USA) were examined by a combination of EDX, EBSD and APT for nano- and atomic-scale analysis of chemical and morphology changes in secondary phase precipitations (SPPs) and alpha-Zr matrix. The results will be compared with similar experiments on unirradiated ZIRLO control samples, revealing coarsened SPPs and segregated nano-layers formed by elements migrating towards energetically favourable locations under irradiation. A discussion will also be presented on how such results contribute to better understanding the disparity of degradation behaviours between ion/proton- and neutron-irradiated materials.

[1] G. He, et al., Investigating the stability of second phase particles in Zr-Nb alloys under irradiation. *Journal of Nuclear Materials*, 2019. 526.

[2] A. Harte, et al., The effect of matrix chemistry on dislocation evolution in an irradiated Zr alloy. *Acta Materialia*, 2017. 130: p. 69-82.

[3] A. Harte, et al., Nano-scale chemical evolution in a proton-and neutron-irradiated Zr alloy. *Journal of Nuclear Materials*, 2017. 487: p. 30-42.

Nano-scale characterization of Nuclear and Radioactive Materials

P81

AN ANALYSIS OF T91 CORROSION IN LEAD BISMUTH EUTECTIC BY CORRELATING APT WITH ELECTRON MICROSCOPY

Minyi Zhang¹, Guanze He¹, Mark Lapington¹, Weiyue Zhou², Michael P. Short², Paul A.J. Bagot¹, Felix Hofmann¹, Michael P. Moody¹

¹University of Oxford, United Kingdom

²Massachusetts Institute of Technology, USA

Lead-bismuth eutectic (LBE) cooled fast reactors, as one of the designs for GenIV reactors promising higher efficiency and improved safety, are drawing increasing attention. T91 steel is one of the commonly-proposed candidate structural materials for these reactors. In this work we examine such T91 materials, prepared with a martensitic microstructure and with carbides and nitrides present to further improve the mechanical properties. In T91, it is desired to improve the corrosion-resistance in LBE environments, which requires a detailed understanding of the corrosion mechanisms. We show by EDX that corrosion of T91 in LBE causes Cr depletion from the matrix, along with the disappearance of Cr precipitates from the sub-surface region. Extracting specimens from this same region for APT confirms the Cr depletion tendency at the atomic scale. Furthermore, by using STEM-EELS, the Cr precipitates present are shown to be carbides, while vanadium nitrides were also detected. APT investigations targeting these two types of precipitates have afforded an understanding of the 3D shape of these and their precise chemical compositions. STEM-EDX cross-sectional scans across the lead corroded, Cr-depleted and matrix regions show that Cr carbides dissociate in the Cr-depleted region, reappearing somewhat larger in the adjacent area before recovering to their normal size deeper in the matrix. Thus, we propose that Cr carbides dissociate in the Cr-depleted region, with the released Cr moving to the LBE layer while carbon diffuses in the opposite direction, deeper towards the unaffected region where it combines with Cr forming larger carbides. Finally, from an EBSD examination of the corroded samples, we observe a grain morphology change in the Cr depleted region, which suggests that the Cr depletion promotes a phase change. The Cr disappearance may reduce the strain in martensite grains, providing the possibility for the martensite to recrystallize. Such a recrystallization will change the surface grain structure, reducing its strength but the toughness will improve, which may be useful for reactor design. Overall these results therefore demonstrate the complexity in behaviour for T91 exposed to LBE environments, along with the need for detailed high-resolution characterisation to improve components in GenIV reactors.

Nano-scale characterization of Nuclear and Radioactive Materials

P82

CORRELATING THE EVOLUTION OF ATOMIC-SCALE MICROSTRUCTURE WITH DETERIORATION OF MECHANICAL PROPERTIES IN IRRADIATED T91 STEEL

Hannah Jones¹, Michael Moody¹, Paul Bagot¹, Anna Kareer¹

¹University of Oxford, United Kingdom

T91 steel is a promising candidate material for high-dose structural components in both fusion and fission nuclear reactors. However, a better understanding of how the atomic-scale microstructure evolves under the extreme temperature and irradiation conditions within the reactor and correlating this to the deterioration of mechanical properties, is still needed to predict safe operating lifetimes of the component. This work combines the 3D atomic-scale microscopy provided by atom probe tomography with micro-mechanical testing by nanoindentation to systematically investigate the effects of irradiation exposure in a set of neutron and ion-irradiated T91 samples provided by the University of Michigan. Analysis of nanoscale clusters found in atom probe data of the samples is used to assist in modelling irradiation hardness as a function of irradiation temperature, and the use of dual ion beam irradiation as a surrogate for neutrons to facilitate a wider range of dose and temperature effects in this material will also be discussed.

Nano-scale characterization of Nuclear and Radioactive Materials

P83

INFLUENCE OF NICKEL ON THE FORMATION OF MN-, NI- AND SI-RICH PRECIPITATES IN REACTOR PRESSURE VESSEL STEELS

Katharina Sophia Ungermann¹, Paul Bagot¹, Christina Hofer¹, Anna Kareer¹, Nick Riddle², Michael Moody¹

¹University of Oxford, United Kingdom

²Rolls Royce, United Kingdom

The Reactor Pressure Vessel (RPV) is one of the integral components for operational safety in nuclear power stations. In pressurized water reactors the RPV is made from low-alloyed bainitic steel. Its material properties degrade over time due to temperature- and irradiation-induced embrittlement. Many studies have reported that the formation of a high number density of Mn-Ni-Si-rich features after exposure to neutron irradiation [1–3] is responsible for the hardening and embrittlement of RPV steels. Understanding the mechanisms influencing the formation of these precipitates in RPV steels subject to irradiation is crucial for the accurate prediction of property changes during operational life. In addition, given that lifetime extensions of nuclear power plants of up to 80 years are now a major discussion point, it is even more important to investigate. To this end, the influence of specific elements, and their relative concentration within the material, on the formation of these precipitates is studied using a spectrum of neutron-irradiated RPV steels with systematically varied alloy composition. In this talk, the influence of the Ni-content on the main driving mechanism behind cluster evolution is explored. Atom Probe Tomography enables us to study such small-scale precipitates in detail, identifying microstructural characteristics as well as elements aiding the formation of these precipitates. Results from this ongoing study show that the number density of precipitates, their composition and their main nucleation sites change with the Ni-content in synergy with other elements.

[1] N. Almirall, P. B. Wells, T. Yamamoto, K. Wilford, T. Williams, N. Riddle, and G. R. Odette, *Acta Mater* 179, 119 (2019).

[2] G. R. Odette, N. Almirall, P. B. Wells, and T. Yamamoto, *Acta Mater* 212, 116922 (2021).

[3] B. M. Jenkins, J. O. Douglas, N. Almirall, N. Riddle, P. A. J. Bagot, J. M. Hyde, G. R. Odette, and M. P. Moody, *Materialia* 11, 100717 (2020).

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P84

ATOM PROBE ANALYSIS OF POLYMER NANOCOMPOSITES

James O. Douglas¹, Stella Pedrazzini¹, Emilio Martínez-Pañeda¹, Reza Salehiyan², Łukasz Figiel³

¹*Imperial College London, United Kingdom*

²*Edinburgh Napier University, Scotland*

³*University of Warrick, United Kingdom*

APT analysis of polymers has been relatively limited compared to many other material systems due to sample preparation, data collection and data analysis challenges. Polymers are often beam sensitive and Focused Ion Beam (FIB) sample preparation is often avoided, leading to approaches based on spray deposition of polymer films [1] or self assembling monolayers [2] onto pre-fabricated sharpened supports. Mass spectra collected from polymers can consist of carbon compound ions, and halogens used in polymers such as chlorine or fluorine are difficult to ionise and so compositional analysis can also be challenging [3]. In this work we present initial analysis of boron nitride 2D nanoparticles (NP) within a thermoplastic semi-crystalline polymer matrix (polyvinylidene fluoride (PVDF)), so called polymer nanocomposite (PNC) – a system considered as a promising material candidate for gas barrier applications. PNC samples were prepared using a melt-compounding process at different NP loadings, injection moulded into discs and characterised using thermogravimetric analysis, differential scanning calorimetry and scanning electron microscopy, alongside gas permeation studies at the macroscopic level. APT site specific FIB sample preparation was then carried out using a Helios Hydra plasma FIB at room temperature. Care was taken to improve bulk sample conductivity with conductive coatings and to reduce beam damage but no additional stages, such as cryogenic sample preparation, were required. Nanoscale regions predicted to be linked to the nanoparticles within the matrix were identified during tip sharpening through secondary electron (high contrast in tip) and backscatter imaging (low contrast in tip). Laser pulsed analysis using a LEAP 5000XR of these samples produces a mass spectra that contains various compound ions linked to fragments of the polymer chamber. In the samples predicted to contain nanoparticles, localised regions that contained boron species were observed along with various compound ions of carbon, boron and fluorine. This work demonstrates the viability of room temperature sample preparation and APT analysis of polymer nanocomposites and the potential for the nanoscale analysis of the particle/matrix interfaces within polymer-based systems.

[1] *Journal of Microscopy*, 2010, 237,2 pp. 155–167

[2] *Langmuir* 2010, 26, 8, 5291–5294. [3] *Langmuir* 2012, 28, 1, 56–59.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P85

ANALYSIS OF WATER FROST USING NEAR AND EXTREME ULTRAVIOLET PULSED ATOM PROBE TOMOGRAPHY

*Jacob Garcia*¹, *Benjamin W. Caplins*¹, *Luis Miaja-Avila*¹, *Norman A. Sanford*¹, *Daniel E. Perea*², *Ann N. Chiaramonti*¹

¹*National Institute of Standards and Technology, USA*

²*Pacific Northwest National Laboratory, USA*

Cryogenically frozen materials with water ice serving as a matrix are of increasing interest for atom probe tomography (APT) analysis, potentially allowing for 3D sub-nm resolution of complex biological materials in a natural hydrated state.[1, 2] Although there has been progress on the underlying mechanisms of field evaporation of frozen water,[3] the production of ice and frost on the sample surface is a common contaminant during cryogenic sample preparation and transfer that requires further characterization. A careful analysis of frost would strongly aid in the analysis of cryogenically transferred samples – especially in the case of ice-based samples. In this presentation, we demonstrate a simple method to produce frosted APT samples that does not require specialized transfer equipment. The analysis of frost under various extreme ultraviolet (EUV) and near ultraviolet (NUV) pulsed APT conditions will be described. Comparisons between the experimental conditions, number of water fragments in the mass spectra, peak shapes, background, number of fragmentation and dissociation peaks, and multiplicity of detection events will be discussed. We will show preliminary data related to the differences in ice versus frost using both EUV and NUV APT systems. A better understanding of frost run with laser-pulsed APT will lead to more accurate cryogenic sample analysis.

[1] P. Stender *et al.*, “Status and Direction of Atom Probe Analysis of Frozen Liquids,” *Microscopy and Microanalysis*, vol. 28, no. 4, pp. 1150–1167, Aug. 2022, doi: 10.1017/S1431927621013994.

[2] I. E. McCarroll, P. A. J. Bagot, A. Devaraj, D. E. Perea, and J. M. Cairney, “New frontiers in atom probe tomography: a review of research enabled by cryo and/or vacuum transfer systems,” *Mater Today Adv*, vol. 7, no. 100090, pp. 1–11, Sep. 2020, doi: 10.1016/j.mtadv.2020.100090.

[3] N. Segreto *et al.*, “Understanding the Underlying Field Evaporation Mechanism of Pure Water Tips in High Electrical Fields,” *J Phys Chem A*, Aug. 2022, doi: 10.1021/acs.jpca.2c04163.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P86

EVAPORATION BEHAVIOUR OF FROZEN LIQUIDS STUDIED BY CRYO-ATOM PROBE TOMOGRAPHY

Kuan Meng¹, Alexander M. Oetken¹, Sakshi Sinha¹, Patrick Stender¹, Guido Schmitz¹

¹University of Stuttgart, Germany

Atom Probe Tomography (APT) has become a mature analysis technique for various materials. The technique is able to deliver high-quality, three-dimensional atomic maps with high spatial and chemical resolution. Nevertheless, the measurement of soft matter and liquids is a tedious task and only a limited amount of data is available today. However, the progress in recent years due to the development of cryo-FIB preparation techniques is remarkable. The observed complexity of the datasets simultaneously presents potential problems, challenges, and opportunities. In this work, we will present our ongoing efforts to overcome the last material barrier for APT analysis. The influence of various parameters on the pure water system, glucose solution and other frozen liquids is discussed. Especially the influence of the parameters, which are easily accessible to the experimenter (effective field strength, laser energy, laser wavelength and measurement frequency), are considered, as far as determined, for the different systems. As well as the techniques used for the preparation of the samples.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P87

UNDERSTANDING THE INFLUENCE OF NANOSCALE SEGREGATION ON THE OSTEOCLAST RESORPTION OF B-TRICALCIUM PHOSPHATES

Selase Torkornoo¹, Marc Böhner², Ingrid McCaroll¹, Baptiste Gault¹, Guido Schmitz¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

²RMS Foundation, Switzerland

Due to excellent biocompatibility and high resorption properties, β -tricalcium phosphate (β -TCP, $\text{Ca}_3(\text{PO}_4)_2$) has become a highly sought after substitute for bone grafts. Yet, the driving force behind the biological responses between β -tricalcium phosphate and osteoclasts remains unexplained at the nanoscale, particularly at grain boundaries. Current studies indicate that phase compositions are not the only factor that influence biological responses (Böhner, 2020). In fact, Le Gars Santoni et. al. (2022) observed that high osteoclastic resorption at grain boundaries occurred within a specific range of Ca/P molar ratios, and that these ratios differed depending on the sample preparation method. In order to better understand the effect of grain boundaries on the osteoclastic resorption, we evaluate the chemical distribution of calcium and impurities at β -TCP grain boundaries using atom probe tomography (APT). This APT study will detail the methods used to accurately quantify the composition of bioceramic materials and elucidate the contributing factor for the osteoclastic resorption at some β -TCP grain boundaries.

[1] Böhner, M., Le Gars Santoni, B. & Döbelin, N. (2020). B-tricalcium phosphate for bone substitution: Synthesis and properties. *Acta Biomaterialia*, 113, 23–41. <https://doi.org/10.1016/j.actbio.2020.06.022>

[2] Le Gars Santoni, B., Niggli, L., Dolder, S., Loeffel, O., Sblendorio, G. A., Heuberger, R., Maazouz, Y., Stähli, C., Döbelin, N., Bowen, P., Hofstetter, W., & Böhner, M. (2022). Effect of minor amounts of β -calcium pyrophosphate and hydroxyapatite on the physico-chemical properties and osteoclastic resorption of β -tricalcium phosphate cylinders. *Bioactive Materials*, 10, 222–235. <https://doi.org/10.1016/j.bioactmat.2021.09.003>

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P88

A NEAR ATOMIC-SCALE VIEW OF THE COMPOSITION OF FERRITIN IN THE HYDRATED STATE BY ATOM PROBE TOMOGRAPHY

Shuo Zhang¹, Leonardo Shoji Aot¹, Tim M. Schwarz¹, Mahander Pratap Singh¹, Eric Woods¹, Mahander Pratap Singh¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

Understanding the 3D structures and chemical nature of biological macromolecules is crucial for deciphering their functions, and developing novel biochemical compounds and processes. In this regard, atom probe tomography (APT) is a promising approach to investigate their chemical compositions with a sub-nanometre resolution. In this study, we aim to analyze ferritin in its hydrated state by APT. A solution containing ferritin particles was deposited on a nanoporous brass substrate, and plunge frozen in liquid nitrogen. We used advanced cryogenic sample preparation techniques including the cryo-(Ga) FIB lift-out to prepare needle-shaped specimens. During the lift-out process, the (Cr) redeposition method was used for welding before the annular milling, followed by transferring the fabricated ice needles to the APT analysis chamber under a low vacuum ($<10^{-7}$) at a cryogenic temperature (< -178 °C). We obtained mass spectra and 3D APT chemical maps, from the solution and the interface between the liquid and metal. Our results show that sodium is distributed throughout the ice needle, with higher concentrations at the metal/liquid interface. Our findings demonstrate the feasibility of using APT to analyze hydrated biological specimens at a near-atomic scale by using frozen solution as a carrier.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P88

A NEAR ATOMIC-SCALE VIEW OF THE COMPOSITION OF FERRITIN IN THE HYDRATED STATE BY ATOM PROBE TOMOGRAPHY

Shuo Zhang¹, Leonardo Shoji Aot¹, Tim M. Schwarz¹, Mahander Pratap Singh¹, Eric Woods¹, Mahander Pratap Singh¹

¹Max-Planck-Institut für Eisenforschung GmbH, Germany

Understanding the 3D structures and chemical nature of biological macromolecules is crucial for deciphering their functions, and developing novel biochemical compounds and processes. In this regard, atom probe tomography (APT) is a promising approach to investigate their chemical compositions with a sub-nanometre resolution. In this study, we aim to analyze ferritin in its hydrated state by APT. A solution containing ferritin particles was deposited on a nanoporous brass substrate, and plunge frozen in liquid nitrogen. We used advanced cryogenic sample preparation techniques including the cryo-(Ga) FIB lift-out to prepare needle-shaped specimens. During the lift-out process, the (Cr) redeposition method was used for welding before the annular milling, followed by transferring the fabricated ice needles to the APT analysis chamber under a low vacuum ($<10^{-7}$) at a cryogenic temperature (< -178 °C). We obtained mass spectra and 3D APT chemical maps, from the solution and the interface between the liquid and metal. Our results show that sodium is distributed throughout the ice needle, with higher concentrations at the metal/liquid interface. Our findings demonstrate the feasibility of using APT to analyze hydrated biological specimens at a near-atomic scale by using frozen solution as a carrier.

Quantification in APT, Interlaboratory Studies, Calibration, Standards

P89

QUANTIFICATION OF ISOTOPIC RATIOS IN ATOM PROBE TOMOGRAPHY: EXAMPLES FROM GEOMATERIALS

David Saxey¹

¹*Curtin University, Australia*

For some geological applications of atom probe tomography (APT) accurate isotopic quantification is a major objective. This is of particular importance when examining nanoscale reservoirs of stable and radiogenic isotopes that may yield information on past geological processes. While there are limitations in the quantitative analysis of samples by APT, there are also ways to mitigate these limitations by optimising the acquisition conditions, or by applying the most appropriate methods for data analysis. This presentation discusses the challenges of quantifying peaks in APT mass spectrum data, covering aspects such as mass ranging, peak shape considerations, and background correction. Examples will be given from isotope geochemistry, where the accuracy of isotopic quantification is especially important, and where comparisons can be made with well-established reference materials. Drawing on extensive studies by Meisenkothen et al. [1] and Prosa & Oltman [2], and previous applications of APT to geological studies [3,4], we will present results from recent work on several geomaterials and reference samples. The ultimate aim is to provide a robust method for isotopic ratio measurement that does not depend heavily on user-selection of mass ranging or free parameters relating to the background correction.

[1] Meisenkothen, F., Samarov, D. V., Kalish, I., & Steel, E. B. (2020). Exploring the accuracy of isotopic analyses in atom probe mass spectrometry. *Ultramicroscopy*, 216, 113018.

[2] Prosa, T. J., & Oltman, E. (2022). Study of LEAP® 5000 Deadtime and Precision via Silicon Pre-Sharpener-Microtip™ Standard Specimens. *Microscopy and Microanalysis*, 28(4), 1019–1037.

[3] Fahey, A. J., Perea, D. E., Bartrand, J., Arey, B. W., & Thevuthasan, S. (2016). Uranium isotopic ratio measurements of U₃O₈ reference materials by atom probe tomography. *Journal of Environmental Radioactivity*, 153, 206–213.

[4] Gopon, P., Douglas, J. O., Meisenkothen, F., Singh, J., London, A. J., & Moody, M. P. (2022). Atom Probe Tomography for Isotopic Analysis: Development of the ³⁴S/ ³²S System in Sulfides. *Microscopy and Microanalysis*, 28(4), 1127–1140.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P90

SUGGESTION FOR COMMUNITY-DRIVEN DATA SCHEMAS TOWARDS INTEROPERABLE REPRESENTATION OF DATA ANALYSES IN ATOM PROBE

Markus Kühbach¹, Sandor Brockhauser¹

¹*Humboldt-Universität zu Berlin, Germany*

Atom probe tomography and related field-ion microscopy became mature nanoscale structure and composition characterization methods^[1, 2]: A technique to gain a more detailed understanding of microstructure-materials-properties relations and a rich collection of distributed point-cloud datasets for developing new and improving existent data-analysis techniques. Both commercial and community-developed software tools have been developed which address and solve different aspects how point-cloud and composition data can be processed into microstructural features and descriptors for microstructure-material-property relations. With the recent activity towards integrating scripting capabilities in AP Suite^[3], the tool landscape has maybe never been brighter for combining the use of commercial and open-source software^[4]. However, publicly accessible documentation of file formats and other data artifacts remain a key blocker for interoperability. Concepts and terms used across tools differ and would profit from being openly documented in a version-controlled manner. In our contribution, we deliver a rich set of suggestions for a community-maintained data schema repository^[5] being designed to describe the majority of the data processing steps which are typically performed in atom probe research including reconstruction and ranging, microstructural feature reconstruction and meshing, point-to-object distancing, and logical correlating of features.

[1] B. Gault et al., *Nat. Rev. Methods Primers* 1 (2021), <https://doi.org/10.1038/s43586-021-00054-x>

[2] S. Katnagallu et al., *New J. Phys.* 21 (2019), <https://doi.org/10.1088/1367-2630/ab5cc4>

[3] <https://github.com/CamecaAPT>

[4] <https://gitlab.mpcdf.mpg.de/nomad-lab/nomad-remote-tools-hub/-/blob/develop/docker/software-list-b5-atom-probe-microscopy.md>

[5] <https://fairmat-nfdi.github.io/nexus-fairmat-proposal>

[6] <https://gitlab.com/paraprobe/paraprobe-toolbox> All webpages were accessed May, 8rd 2023 the last time.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P91

ESTIMATION OF ROOM-TEMPERATURE DIFFUSIVITY OF ZN IN AL BY ATOM PROBE TOMOGRAPHY AND CAHN-HILLIARD THEORY

Xinren Chen¹

¹*Max-Planck-Institut für Eisenforschung GmbH, Germany*

During the natural aging of aluminum alloys, the rates of atomic clustering and precipitation strengthening are closely related to the atomic diffusivity at room temperature. However, the measurement of the equilibrium room-temperature diffusivity of solutes is a long-standing technique problem due to the small diffusion distance of atoms. Another well-known problem is the remaining excess vacancies in the system interfere with the measurements. In this work, we demonstrate a low-temperature diffusivity measurement method that is based on the kinetic assessment of the spinodal decomposition process by atom probe tomography, which lowers the requirement for the diffusion distance for diffusivity measurements. The nano aging experiment was adopted to eliminate the influence of excess vacancies. The results give a tracer diffusivity of Zn in the Al-12.5 at.%Zn alloy at 295 K. The work provides a new pathway for getting diffusivity data at low temperatures by using atom probe tomography and spinodal decomposition, which can be widely applied to the systems with spinodal decomposition.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P92

INVESTIGATING QUANTIFICATION BIAS IN LREEPO

Tom Veret¹

¹University of Rouen Normandie, France

Monazite is a mineral often used in geochronology as a geo-chronometer because of its ability to store elements such as uranium and thorium during its crystallisation. Laser-assisted atom probe tomography (LA-APT) measurements of the chemical composition of such minerals provide access to isotopic information on a scale never before available and allow the identification of nanoscale chemical heterogeneities that provide insight into the geological and thermal history of terrestrial and extraterrestrial rocks [1]. However, biases in the isotopic quantification of these materials (~2-3 At%), which are still poorly understood at present, lead to significant dating errors. Phosphates have very similar structures to monazite (LREE*PO₄) and are therefore ideal candidates to study the impact of analytical parameters on the quantitative isotopic measurement of different rare earths. Different approaches have been proposed in the literature to understand the causes of these measurement uncertainties on materials of interest. Firstly, laser characteristics such as wavelength (nm) and power (pJ) coupled with the effect of the electric field (V/nm) can lead to evaporation phenomena (co-evaporation, multiple shots, dissociation) or data processing problems (pile-up, equal m/n ratios) that cause counting errors and therefore biases on the isotopic quantification of elements [2]. In addition, the analysis of non-metals in APT often results in evaporation due to the effect of molecular ion fields, resulting in mass spectra with "forest of peaks" that make identification very complex. The implementation of decomposition and deconvolution procedures [3] would make it possible to deal with peak interference while making identification more accurate and repeatable in order to obtain at least semi-quantitative composition measurements. *Light Rare Earth Element.

[1] Steven M. Reddy et al. « Atom Probe Tomography: Development and Application to the Geosciences ». *GEOSTANDARDS and GEOANALYTICAL RESEARCH*, 44, 5-50, 2019.

[2] Dhamodaran Santhanagopalan et al. « Effects of laser energy and wavelength on the analysis of LiFePO₄ using laser assisted atom probe tomography ». *Ultramicroscopy* 148, 57-66, 2014.

[3] Frederick Meisenkothen et al. « Exploring the accuracy of isotopic analyses in atom probe mass spectrometry ». *Ultramicroscopy* 216, 113018, 2020.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P93

TOWARDS A REDUCTION OF SELECTIVE LOSSES IN ATOM PROBE TOMOGRAPHY EXPERIMENTS

Christian Bacchi¹

¹University of Rouen Normandie, France

In 2005, an APT detection system, called the Advanced Delay Line Detection system (aDLD), emerged and promised an improvement of the multi-hit capacity of APT detectors [1]. With the help of fast digitizers, the aDLD system has the ability to decompose DLD output signals, potentially overlapped during multiple events, into individual signals. Through this development, it has been demonstrated that the dead-time of conventional APT detection systems, reaching around 3 ns [2], could be reduced to 1.5 ns; which theoretically help to reduce compositional biases from APT analyses [3]. Results from APT instruments equipped with the aDLD system show that compositional biases may still be significant with this lower dead-time [4-5]. This can be explained by the fact that a high proportion of losses are still concentrated in a time and space region around primary ion-impacts where secondary ion-impacts cannot be resolved [6]. Given that the aDLD algorithm for decomposing overlapped signals is mainly based on the recognition of signal slopes [1], it becomes difficult to separate highly overlapped signals due to resulting signal envelopes having the same slopes as signals from single events. It may be found that, despite the shape similarity between highly overlapped signals and individual signals from single events, it is still possible to discriminate them through the computation of the amount of charge deposited on the delay lines. Consequently, the association of the aDLD shape recognition with the computation of charge quantities turns out to be useful for decomposing overlapped signals with time differences under the standard aDLD dead-time of 1.5 ns. This new advanced detection system, named ChaSe (Charge Sensitive) DLD system, promises the improvement of spatial and compositional performances of APT instruments through a new set of algorithms using the charge sensitivity of DLDs.

- [1] G. Da Costa et al., *Review of Scientific Instruments* 76 (1) (2005): 013304
- [2] Z. Peng et al., *Ultramicroscopy* 189 (2018): 54-60.
- [3] G. Da Costa, et al., *Review of Scientific Instruments* 83.12 (2012): 123709.
- [4] T. Kinno, et al., *Applied surface science* 259 (2012): 726-730.
- [5] R. Cuduvally et al., *Ultramicroscopy* 210 (2020): 112918.
- [6] C. Bacchi et al., *Microscopy and Microanalysis* 25.2 (2019): 418-424.

Nano-scale characterization of Biological and Organic Materials, Frozen Liquids, Soft Matter

P94

ASSESSING THE EFFECTIVE SPATIAL PERFORMANCE OF ATOM PROBE TOMOGRAPHY

Frédéric De Geuser¹, Baptiste Gault¹

¹Max-Planck Institut für Eisenforschung GmbH, Germany

²Imperial College London, United Kingdom

When assessing the quality of atom probe tomography 3D reconstructions, one faces several challenges largely related to the fact that APT is unlike classical microscopy techniques. The physics underlying field evaporation of the ions, the instrumental configuration, the sample geometry and composition, etc... are all factors that make the spatial resolution not only difficult to predict but also heterogeneous across the dataset. In fact, one could argue that this heterogeneity prevents any useful definition of the spatial resolution of APT (or even of an APT experiment/dataset). But more than a vocabulary issue, the most prominent challenge in assessing the spatial performance of APT is finding adequate reference or calibration standards. The 3d analytical imaging capabilities of single objects are so unique that there generally exists essentially no technique that could validate our reconstruction at the atomic level. The only exception so far was the use of atomic planes resolving power as a reference for spatial resolution in the vicinity of crystallographic planes. This however is very restrictive in that it limits the method to i) datasets where planes can be resolved, ii) areas close to poles and iii) spatial resolution in particular directions. It is also a very ideal measure of the spatial performance and is a very poor measure of the effect of the reconstruction on e.g. the position of 2 neighbouring atoms or the detection of a small cluster. Recently, 2 experimental techniques have been suggested to serve as a reference for assessing the spatial performance of APT. One is *in situ*, so to speak, since it involves field ion microscopy (FIM) during APT acquisition. The other is small-angle X-ray scattering (SAXS) which is also sensitive to chemical fluctuations at the atomic level with no resolution issues. With their help, we will discuss the spatial performance of APT when it comes to quantifying particles/clusters or solute environments in solid solutions with examples from several diluted structural alloys or concentrated alloys akin to high-entropy alloys.

Nano-scale characterization of Functional materials, Battery materials, Ceramics, Oxides

P95

METAVALENT BONDING IN LAYERED CHALCOGENIDES REVEALED BY ATOP PROBE TOMOGRAPHY

Jan Köttgen¹, Anna Vymazalová², Yuan Yu¹, Matthias Wuttig¹

¹ *RWTH Aachen University, Germany*

² *Czech Geological Survey, Czech Republic*

Metavalent bonding (MVB) is a unique bonding mechanism that differs significantly from metallic, ionic and ordinary covalent (2 center – 2 electron) bonding. MVB describes a situation where adjacent atoms share half an electron pair (one electron) to form a σ -bond. This bonding mechanism enables a unique property portfolio such as a high Grüneisen parameter, a large optical dielectric constant, and a high Born effective charge. These properties lead to promising applications of MVB compounds, such as GeTe or Sb₂Te₃, for phase change memory and thermoelectric energy conversion. Besides these property-based fingerprints, MVB shows an abnormal bond-rupture behavior with a high probability to dislodge several fragments, i.e., the probability of multiple events (PME), in atom probe tomography. Given these special properties which ensure broad application potentials in many electronic and optical fields, it is of great significance to explore more MVB candidates. In this project, we want to expand our investigations in the fields of layered transition metal chalcogenides such as PdTe₂ and Pt₃Te₄ as well as layered minerals like Vihorlatite (Bi₂₄Se₁₇Te₄) and Hedleyite (Bi₇Te₃). All these layered systems showed a high PME value during the laser-assisted atom probe tomography measurements, indicating these special chemical bonding mechanisms. This paves the way for further investigations to expand our existing understanding of metavalent compounds to materials that are made by nature.

First Name	Last Name	O = Oral P = Poster
Anthony	Akinwale	P61
Gabe	Arcuri	P4
Jonas	Arlt	O11.5
Christian	Bacchi	P93
Paul	Bagot	O5.4, O8.3, O9.2, O12.2, P45, P60, P61, P80, P81, P82, P83
Shalini	Bhatt	J.H. Block Lecture, P23
Torben	Boll	O11.3, P28, P49
Joe	Bunton	O3.5, P63
Alex	Cackett	O5.2, P79
Julie	Cairney	O7.1, O10.1, P55, P71
Anne	Callow	P76
Megan	Carter	O9.5
Ann	Chiaromonti	O12.3, P5, P85
Oana	Cojocaru-Mirédin	O1.2, P27, P26, P43
Oana	Cojocaru-Mirédin	O1.2, P43, P26, P27
Shelly	Conroy	O10.4
Calum	Cunningham	O5.2, P79
Michal	Dagan	P60
Alexander	Dahlström	O4.3, P51
Frédéric	De Geuser	P94
Karen	DeRocher	O12.5
Arun	Devaraj	O7.2, O10.5
Aissatou	Diagne	P29, P72, P73
Masoud	Dialameh	O2.2, P25
James	Douglas	O1.5, O7.4, O10.4, P84
Sebastian	Eich	O6.2, O9.4, P39
Gustav	Eriksson	O10.3
Florant	Exertier	P12
James	Famelton	P45, P61
Andrea	Fazi	P32, P34
Peter	Felfer	O3.2, O6.4, O13.1, P36
Claudia	Fleischmann	O2.2, O12.4, P14, P15, P66, P67
Richard	Forbes	P20, P21, P22
Martí López	Freixes	EM1
Christoph	Freysoldt	J.H. Block Lecture, O6.3, P23, P24, P65
Jacob	Garcia	O12.3, P5, P85
Baptiste	Gault	EM4, O5.1, O6.3, O7.4, O10.1, O10.2, O10.4, O11.4, O11.6, O13.3, O14.3, P19, P24, P33, P37, P38, P39, P60, P68, P87, P88, P94
Stephan	Gerstl	P11
Sonia	Guehairia	P50
Sonia	Guehairia	P50
Ezgi	Hatipoglu	O11.6
Martina	Heller	O6.4, P70
Christina	Hofer	O12.2, P76, P83
Tobias	Hofmaenner	P3
Natalie	Holmes	O7.1

Coraline	Hossepied	P2
Rong	Hu	P77, P78
Severin	Jakob	O9.1, O13.2
Anna	Jelinek	P54
Benjamin	Jenkins	O14.2
Zheng	Jianshu	O6.2
Hannah	Jones	P82
Takahahshi	Jun	O2.3
Uzuhashi	Jun	O3.4
Azam	Karami	P26, P27
Shyam Swaroop	Katnagallu	J.H. Block Lecture, O6.3, P24
Heena	Khanchandani	P69, O13.3
Yoon Jun	Kim	P48
Sebastian	Koelling	O14.1
Jan	Köttgen	P95
Mathias	Krämer	P38
Vitaly	Krasnov	P67
Norbert	Kruse	O2.1
Markus	Kühbach	O6.1, P65, P90
David	Larson	O3.3, P63, Plenary Lecture, P10
Yue	Li	O14.3
Jhao-Rong	Lin	P66
Xiaohuan	Liu	O4.5
Fang	Liu	P33
Marc	Maier	P3
Jean-Baptiste	Maillet	P74
Ross	Marceau	O7.3
David	Mayweg	O5.3, P13
Ingrid	McCarroll	EM4, O10.1, P55
Davit	Melkonyan	O4.2
Kuan	Meng	O7.5, P86
Luis	Miaja-Avila	O12.3, P5, P85
Michael	Moody	O1.5, O9.2, O9.5, O12.2 P42, P45, P60, P61, P80, P81, P82, P83
Richard	Morris	O2.2, O12.4, P14, P66
Isabelle	Mouton	O5.1
Varatharaja	Nallathambi	P39
Samba	Ndiaye	P29, P72, P30
Nicole	Nevill	EM2
Sieglind	Ngai	O9.3
Sylvain	Nulli	P56
Benedict	Ott	O6.4, P21, P35, P70
Michael	Pavel	O9.3, P59
Amanda	Persdotter	P35
Emily	Peterman	O8.1
Nikita	Polin	O11.4
Jonathan	Poplawsky	O1.3, O10.5

Ivan	Povstugar	O1.4, O11.2
K.G.	Pradeep	O4.4, P46
Mahander	Pratap Singh	O7.4, P33, P37, P88
Sophie	Primig	O4.1, O9.6
Ty	Prosa	O3.3, P9, P10
Bertrand	Radiguet	O5.5, O14.2
Julia	Rau	P32
Steven	Reddy	O8.1, O8.2, O8.3, EM2
Alexander	Reichmann	O6.1
David	Reinhard	O3.3, P6, P9, P10, P63, P64
Anicha	Reuban	O1.4
Katherine	Rice	P9
Lorenzo	Rigutti	P29, P31, P17, P72, P12, P73, P30
Kieran	Rivers	O5.4
Jan Erik	Rybak	P19
Murali	Sadhasivam	P46
Aparna	Saksena	O11.6, O13.3, P52, P65
David	Saxey	EM2, O8.1, O8.2, O8.3, O8.4, P89
Jeroen	Scheerder	O2.2, O12.4, P14, P15, P66, P67
Guido	Schmitz	O6.2, O7.5, O9.4, P44, P86
Tim M.	Schwarz	EM4, O11.5, P19, P68, P88
Hossein	Sepehri Amin	O1.1
Gang	Sha	P47, P77, P78, O4.5
Won-Sang	Shin	P48
Ruiying	Shu	O12.2
Sakshi	Sinha	P86
Nadya	Spettel	P15
Victoria	Strutt	O9.2
Paul	Styman	O5.2, P79
Levi	Tegg	O10.1, P55
Gregory	Thompson	P57, P58
Mattias	Thuvander	O5.3, O9.1, O12.4, O13.2, P13, P32, P34
Selase	Torkornoo	P87
Rob	Ulfig	P10
Katharina Sophia	Ungermann	P83
Paul	van der Heide	P67
Hugo	van Leeuwen	O3.5
André	Vantomme	O2.2, P15, P66
Rick	Verberne	O8.2, P12
Tom	Veret	P92
Vitor	Viera Rielli	O9.6
Thierry	Visart de Bocarmé	O2.1
Florian	Vogel	O9.3
Nora	Vorlaufer	P36
François	Vurpillot	P29, P17, P30, P18,
Oliver	Waszkiewicz	O10.2

Mark	Weaver	O9.3, P59
Johannes	Wild	P49
Eric	Woods	P37, P88, EM4, O7.4, O10.1, O10.2
Yinxing	Wu	P47
Min	Wu	P7
Shuo	Zhang	O7.4, P88
Wenyu	Zhang	P80
Minyi	Zhang	P81