

Report of the

79th IUVSTA Workshop

“3D Chemical Imaging – From fundamentals to advancing applications”

Chair: Rasmus Havelund, National Physical Laboratory, United Kingdom

Co-chairs: Claudia Fleischmann, Imec, Belgium
Nataschia De Leo, Istituto Nazionale di Ricerca Metrologica, Italy

Location: Hotel Flamingo Resort, Santa Margherita di Pula, Italy

Dates: 15 - 19 May 2017

IUVSTA division: Applied Surface Science

Website: <http://bit.ly/iuvsta>

Introduction

In the continued innovation in high value-added manufacturing, industry is increasingly using 3D architectures, additive manufacturing and a rapidly-expanding library of materials. This creates a need for beyond state-of-the-art capabilities to measure chemical composition and interfacial properties with 3D-spatial resolution.

The 79th IUVSTA workshop brought together leading scientists in atom probe tomography, focused ion beams, 3D secondary ion mass spectrometry, and related methods in in-depth discussion to identify and solve current challenges and issues related to these methods.

Participants

The workshop had a total of 50 participants of which 12 were invited speakers. The participants represented academia, national laboratories and industry from 11 countries. A list of participants is provided in Appendix A.

Workshop Programme

In addition to the 12 invited contributions, the program comprised 25 contributed talks and 6 poster contributions. These were organised in 12 themed sessions, each covering a topic through, typically, one invited talk and two or three contributed talks followed by a lively discussion session of around one hour chaired by an appointed discussion leader. The scientific programme is included in Appendix B. Workshop participants enjoyed the opportunity to continue the discussions and build new relationships over meals and into the evenings.



The participants at the beautiful Tuerredda beach during the workshop excursion.

Summary of scientific discussion

Key points of the workshop discussions are summarised in three overarching topics below.

3D chemical imaging at the nanoscale using atom probe tomography (APT)

APT offers unique capability for atomic scale reconstruction but in many systems artefacts occur causing reconstructions to be inaccurate spatially and stoichiometrically. Many of the challenges that need to be overcome to further advance APT were discussed during the workshop. A critical point is the evolution of the tip shape as atoms are evaporated from this. The need for improved simulations to support modelling of the tip shape evolution and the implementation of methods for direct measurement of the tip shape were emphasized. It was further discussed that more controlled tip evaporation could be achieved at reduced temperature, and increased detection efficiency, preferably to 100%, would significantly improve accuracy. These are being targeted in current technology projects.

3D chemical imaging using secondary ion mass spectrometry (SIMS)

The applicability of SIMS for 3D imaging has expanded to organic materials with the development of large cluster ion sources that permit sputtering of organics with minimal degradation to the sample surface. This works well for most organic materials but has limited applicability for hybrid organic-inorganic materials due to large differences in sputter rates between different material classes. Sputter rate differences was a recurring topic at the workshop, and it was widely agreed that this is a fundamental challenge.

Three routes to minimising sputter rate differences were discussed: 1) The use of a chemically etching large cluster ion beam to increase the erosion rate of inorganic materials, 2) the use of a high energy small cluster ion beam in conjunction with the use of a radical scavenger to reduce molecular damage caused by the ion beam, and 3) the use of low energy Cs^+ beam for which sputter rates are more uniform. Whilst all these approaches have their merits, none of them fully bridge the significant sputter rate gap that exist between organic and inorganic materials, and all of them introduce new problems. The use of focused ion beams for slice-and-view analysis (FIB-SIMS tomography) was highlighted in several contributions as an emerging alternative for the analysis of hybrid materials.

Related methods and correlative imaging

The workshop covered several related methods including x-ray tomography, electron tomography and FIB-SEM/EDX. Particular emphasis was given to discussions related to correlated imaging where several examples of the importance of this were shown, including the use of TEM reference measurements for improved APT reconstruction and the use of SPM measurements to correct for sputter rate differences and sample topography in 3D SIMS imaging. The workshop identified a need for common data formats and verified methods for bringing together data from different sources.

Acknowledgements

The workshop was made possible through the sponsorship by the International Union for Vacuum Science, Technique, and Applications (IUVSTA). We are also grateful for additional financial support provided by INRIM, ION-TOF GmbH, Ionoptika, and ThermoFisher Scientific which helped keep the delegate rate low.

Financial statement

It is certified that the financial support of €6000 provided by IUVSTA for the 79th IUVSTA workshop was fully used to partially cover fixed costs for 11 invited speakers.

Appendix A: List of participants

Kateryna Artyushkova	University of New Mexico	USA
Jean-Nicolas Audinot	Luxembourg Institute of Science & Technology	Luxembourg
Jean-Paul Barnes	CEA - LETI	France
Jenifer Barrirero	Saarland University	Germany
Paul Blenkinsopp	Ionoptika	UK
Luca Boarino	INRiM	Italy
Eleonora Cara	INRiM	Italy
Giacomo Ceccone	European Commission, Joint Research Centre, Ispra	Italy
Nataschia De Leo	INRiM	Italy
Arnaud Delcorte	Université Catholique de Louvain	Belgium
Masoud Dialameh	INRiM	Italy
Marzia Fantauzzi	Università di Cagliari	Italy
Sarah Fearn	Imperial College London	UK
Peter Felfer	FAU Erlangen-Nürnberg	Germany
Federico Ferrarese Lupi	INRiM	Italy
Gregory Fisher	Physical Electronics	USA
Claudia Fleischmann	Imec	Belgium
John Fletcher	University of Gothenburg	Sweden
Stephan Gerstl	ETH Zurich	Switzerland
Ian Gilmore	National Physical Laboratory	UK
Hubert Gnaser	University of Kaiserslautern	Germany
Dan Graham	University of Washington, NESAC/BIO	USA
Birgit Hagenhoff	Tascon GmbH	Germany
Rasmus Havelund	National Physical Laboratory	UK
Alex Henderson	Manchester University	UK
Anja Henß	Justus-Liebig University Giessen	Germany
Thomas Kelly	CAMECA Instruments	USA
Felix Kollmer	ION-TOF GmbH	Germany
Antonino Licciardello	University of Catania	Italy
Jiro Matsuo	Kyoto University	Japan
Davit Melkonyan	Imec	Belgium
Maiglid Moreno Villavicencio	CEA - LETI	France
Guido Mula	Università di Cagliari	Italy
Johannes Mulders	FEI	The Netherlands
Ewald Niehuis	ION-TOF GmbH	Germany
Cristiana Passiu	ETH Zurich	Switzerland
Lucien Penlap Woguia	CEA - LETI, MINATEC	France
Elisa Pinna	Università di Cagliari	Italy
Frank Uwe Renner	Hasselt University	Belgium
Lorenzo Rigutti	Université de Rouen	France
Marcus Rohnke	Justus-Liebig University Giessen	Germany
Antonella Rossi	Università di Cagliari	Italy
Aleksei Savenko	FEI	The Netherlands
David J. Scurr	Nottingham University	UK
MariaVitalia Tiddia	National Physical Laboratory/Università di Cagliari	UK/Italy
Luigi Antonio Urtis	Università di Cagliari	Italy
Wilfried Vandervorst	Imec	Belgium
Francois Vurpillot	Université de Rouen	France
Tom Wirtz	Luxembourg Institute of Science & Technology	Luxembourg
Andreas Wucher	University of Duisburg-Essen	Germany
Xiao-Ying Yu	Pacific Northwest National Laboratory	USA

Appendix B: Scientific programme

Monday 15th May

8:30: Welcome (Rasmus Havelund)

Session 1: Cluster ion sputtering & 3D SIMS

Discussion leader: Andreas Wucher

8:35: INVITED: Large clusters for 3D SIMS imaging: New physics, new opportunities and... new challenges, *A. Delcorte, V. Cristaudo, R. Edwards, K. Moshkunov, C. Poleunis, E. Pospisilova, S. Surana*

9:10: Characterization and chemical analysis of nanomaterials in complex matrices by ToF-SIMS and XPS, *A. Valsesia, R. La Spina, I. Ojea, I. Rio-Echeverria, P. Colpo, D. Gilliland and G. Ceccone*

9:30: Digging into the challenges in 3D ToF-SIMS Imaging, *Daniel J. Graham and Lara J. Gamble*

Session 2: Data visualization and 3D SIMS/SPM

Discussion leader: Antonino Licciardello

10:55: Strategies for 3D reconstruction from SIMS/SNMS imaging depth profiles, *A. Wucher, T. Heckhoff, C. Heuser, L. Breuer, N. Popszun, N. Winograd*

11:15 Development of 3D high resolution imaging by the correlation of ToF-SIMS and AFM: application to selectively grown GaAs based structures, *Maiglid Moreno, Gael Goret, Denis Mariolle, Franck Bassani, Isabelle Mouton, Brice Gautier, Nicolas Chevalier, Jean Paul Barnes*

11:35: 3D Chemical Analysis of Inorganic and Organic Nanostructures using ToF-SIMS and In-situ SPM, *Ewald Niehuis, Rudolf Moellers, Felix Kollmer, Derk Rading, Henrik Arlinghaus, Andreas Duetting, Raphaelle Dianoux, Adi Scheidemann*

11:55: Combined in-situ Scanning force microscopy/TOFSIMS and FIB-TOFSIMS analysis: Towards 3D chemical analysis, *T. Conard, A. Franquet, V. Spampinato, J. Op de Beeck, U. Celano, V. Vandervorst, R. Moellers*

Session 3: APT prospects, trends and industrial needs

Discussion leader: Ian S. Gilmore

14:30: INVITED: Developmental Prospects for Atom Probe Tomography, *Thomas F. Kelly*

15:05: Fundamental and Applied Aspects of Vacuum-Cryo-Transfer Capabilities for Atom Probe Tomography, *Stephan S.A. Gerstl, Sebastian Tacke*

15:25: INVITED: Industrial application of atom probe tomography to semiconductor devices and manufacturing: dream or reality?, *W. Vandervorst*

Tuesday 16th May

Session 4: FIB tomography I

Discussion leader: Kateryna Artyushkova

8:30: INVITED: FIB-SIMS cross section analysis and tomography of inorganic and organic surfaces, *Felix Kollmer, Rudolf Moellers, Derk Rading, Henrik Arlinghaus, Ewald Niehuis*

9:05: Challenges of 3D FIB-SIMS in materials analysis, *Marcus Rohnke, Christian Schneider and Jürgen Janek*

9:25: SPONSOR CONTRIBUTION: Latest Dual-Beam techniques for sample preparation and 3D analysis, *Aleksei Savenko*

Session 5: FIB tomography II

Discussion leader: Marcus Rohnke

10:55: INVITED: FIB-TOF Imaging & 3D Tomography: Successes and Challenges, *Gregory L. Fisher, David M. Carr, Shin-ichi Iida, Takuya Miyayama and Scott R. Bryan*

11:30: FIB-ToF-SIMS Analysis of Nuclear Waste Glass Material, *Sarah Fearn, Richard Chater, Nor-Ezzaty Ahmad*

11:50: Non-mechanical method for preparation of cross-sections of organic materials for micro chemical analysis using gas cluster ion beam sputtering, *Ichiro Mihara, Rasmus Havelund, Ian S. Gilmore*

Session 6: APT: Accuracy in reconstruction & quantification

Discussion leader: Thomas Kelly

14:30: INVITED: From the quantum scale to the mesoscopic scale: The need of modelling tools in Atom Probe Tomography, *F. Vurpillot, S. Parviainen, D. Zanuttini, B. Gervais*

15:05: INVITED: Atom Probe Tomography of compound semiconductors: compositional accuracy and complementary Transmission Electron Microscopy, *L. Rigutti, L. Mancini, E. Di Russo, F. Moyon, S. Moldovan, C. Hatzoglou, W. Lefebvre, D. Blavette, F. Vurpillot*

15:40: Accuracy in APT analysis: The case of Boron in Silicon, *D. Melkonyan, C. Fleischmann, J. Bogdanowicz, R.J.H. Morris and W. Vandervorst*

(Coffee break 16:00 – 16:20)

16:20: Correlating atom probe tomography and electron tomography of semiconductor devices, *A. Grenier, I. Mouton, G. Audoit, R. Estivill, M. Juhel, S. Duguay, F. Vurpillot, D. Blavette, J-P. Barnes*

Wednesday 17th May

Session 7: Correlative imaging

Discussion leader: Rasmus Havelund

8:30: INVITED: Correlative Microscopy based on Secondary Ion Mass Spectrometry for High-Resolution High-Sensitivity Nano-Analytics, *Tom Wirtz, Jean-Nicolas Audinot, David Dowsett, Santhana Eswara*

9:05: INVITED: Correlative FIB-TOF-SIMS and X-ray nanotomography, *J-P Barnes, A. Priebe, G. Goret, G. Audoit, J. Laurencin, A. Bordes, E. De Vito, P. Bleuet*

9:40: Correlative FIB-TOF-SIMS and X-ray nanotomography, Image Fusion in SIMS-based Correlative Microscopy: Methodology and Applications, *J-Nicolas Audinot, Florian Vollnhals, Santhana Eswara, David Dowsett and Tom Wirtz*

Session 8: Electron tomography, 3D EDX, and MEIS

Discussion leader: Luca Boarino

10:55: 3D-MEIS for structural & compositional characterization of Fin-silicon based nanostructures for sub-22 nm technological nodes, *Lucien Penlap W., F. Pierre, Denis Jalabert, R. Bortollin P., F. Mazen, Dario F. Sanchez, J.P. Barnes*

11:15: Optimization of EDX for local quantification of thin film thickness, *J.J.L. Mulders ThermoFisher Scientific, Eindhoven, The Netherlands*

11:35: FIB-SEM tomography of electrodes for fuel cells: visualization and quantification of 3D structure of porous materials, *Kateryna Artyushkova, Michael Workman, Alexey Serov and Plamen Atanassov*

11:55: Er in porous silicon: where does the light come from?, *Guido Mula, Tony Printemps, Christophe Licitra, Elisa Sogne, Francesco D'Acapito, Narciso Gambacorti, Nicola Sestu, Michele Saba, Elisa Pinna, Daniele Chiriu, Pier Carlo Ricci, Alberto Casu, Francesco Quochi, Andrea Mura, Giovanni Bongiovanni, Andrea Falqui*

Thursday 18th May

Session 9: APT applications – nanoparticles and clusters

Discussion leader: Stephan Gerstl

8:30: Analysis of nanoparticles in the atom probe, *Peter Felfer*

8:50: Nanostructured Si-Based Anode Materials for Li Ion Batteries Studied By Atom Probe, *Y. Zheng, A. Marshal, K.G. Pradeep, F.U. Renner*

9:10: Clustering in crystallographic defects in the eutectic structure of Al-Si casting alloys. Atom Probe Tomography study, *Jenifer Barrirero, Michael Engstler, Naureen Ghafoor, Magnus Odén, Frank Mücklich*

Session 10: SIMS bioimaging

Discussion leader: Daniel J. Graham

10:55: INVITED: Limitless fun! 3D ToF-SIMS analysis in the polyatomic and cluster age, *John S. Fletcher*

11:30: Imaging of Nanoparticles in Tissue Sections: Results of the NanoBioDetect Project, *Birgit Hagenhoff, Lothar Veith, Daniel Breitenstein*

11:50: 3D Chemical Imaging of Skin Tissue, *Nichola J. Starr, Judata Wibawa, Ian Marlow, Mike Bell, David A. Barrett, David J. Scurr*

Session 11: Frontiers in 3D SIMS I

Discussion leader: David J. Scurr

14:30: INVITED: Organic Material Analysis with a SIMS System Equipped with a High-Current Gas Cluster Ion Source and Q-TOF MS/MS, *Jiro Matsuo*

15:05: *In Situ* Chemical Imaging of Biointerfaces Using Microfluidics, *Xiao-Ying Yu*

Poster session (16:20)

ToF-SIMS applied to asbestos characterization, *M. Fantauzzi, C. Passiu, F. Spadaro and A. Rossi*

Lateral Resolution in 3D Imaging of Gold Pillars in a Silver Matrix, performed by means of ToF-SIMS Cesium Depth-Profiling, *Cristiana Passiu, Nicolas Mine, Laetitia Bernard, Nicholas D. Spencer, Antonella Rossi*

Accurate quantification in atom probe tomography reconstruction by correlative electron tomography approach on nanoporous materials, *Isabelle Mouton, Tony Printemps, Adeline Grenier, Pierre Bleuet, Elisa Pinna, Mariavitalia Tiddia, Annalisa Vacca, Guido Mula*

Organic signal recovery using argon cluster ion beams, *Mariavitalia Tiddia, Guido Mula, Ichiro Mihara, Rasmus Havelund, Ian S. Gilmore*

Friday 19th May

Session 12: Frontiers in 3D SIMS II

Discussion leader: Birgit Hagenhoff

9:00: INVITED: 3D imaging using the OrbiSIMS with single beam, dual beam and dual spectrometer modes, *Ian S. Gilmore*

9:35: Digging into Data: Analysis and Visualisation in 3D, *Alex Henderson*

Summary discussion

10:55: Summary of key items, areas of recommended future research, potential collaborations and outlook, *Andreas Wucher, Antonino Licciardello, Ian S. Gilmore, Kateryna Artyushkova, Marcus Rohnke, Thomas Kelly, Rasmus Havelund, Luca Boarino, Stephan Gerstl, Daniel J. Graham, David J. Scurr, Birgit Hagenhoff*

79th IUVSTA workshop
3D Chemical Imaging

From fundamentals to advancing applications

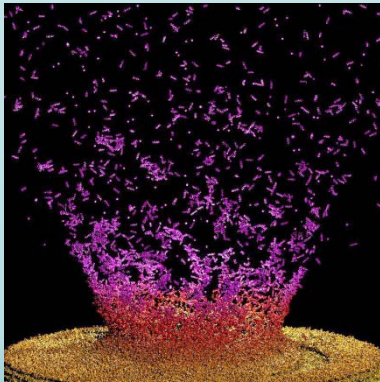
15 – 19 May 2017
Hotel Flamingo Resort
Santa Margherita di Pula, Sardegna



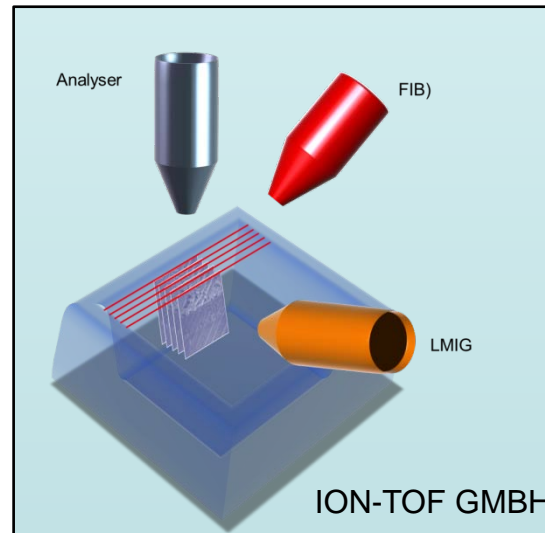
Topics

Cluster ion sputtering and SIMS

- Fundamentals
- Novel ion beams
- 3D reconstruction
- Buried interfaces and defects



Zbigniew Postawa

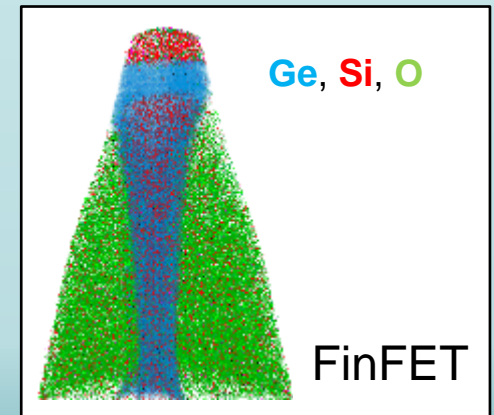


FIB tomography

- Practical analysis
- Minimizing artefacts
- Applications

Atom probe tomography

- Hetero-structures (artefacts)
- Reconstruction
- Applications
- Standardisation



Synergies, complementarities and correlated 3D imaging

Key data

Participants

- 50 participants from 11 countries
- 12 invited speakers

Scientific programme

- 12 themed sessions, each with 35 – 90 min for discussion
- 37 talks and 6 posters



Financial overview

It is certified that the financial support of €6000 provided by IUVSTA for the 79th IUVSTA workshop was fully used to partially cover fixed costs for 11 invited speakers.

Income	Amount (€)
IUVSTA sponsorship	6000.00
Industry sponsorships (three contributors)	4000.00
Registration fees (ex VAT)	12889.32
	22889.32

Costs	Amount (€)
Airport transfer for delegates	2059.20
Venue incl. invited speaker rooms	15809.25
Excursion	4416.80
	22285.25

Note: Not all costs are included in the above

The workshop organising committee thank IUVSTA for the opportunity to organize the workshop which we consider was highly successful in bringing together people for a week of intense scientific discussion of important topics in 3D chemical imaging.

The workshop could not have taken place without the financial support from IUVSTA.

Thank you!

Rasmus Havelund, National Physical Laboratory, UK