
FIFTH
SALVE WORKSHOP
2015

REPORT

The 5th SALVE Summer-Workshop was organized by Prof. Ute Kaiser (Ulm University) and, as previous workshops, held in Hirschegg (Austria) in the Alps, from June 29 to July 1, 2015. The workshop was a meeting of the members of the Sub-Ångstrøm Low-Voltage Electron Microscopy (SALVE) Project Team of Ulm University (25 participants), of the company Corrected Electron Optical Systems GmbH (CEOS) (2 participants), and of the company FEI Electron Optics (1 participant), as well as guests from Dresden University (1 participant) and the University of Heidelberg (1 participant). The program of the workshop included an opening presentation and 15 lectures on various aspects of instrumentation, theory and methods, on radiation damage and on applications of LV HRTEM and as usual a mini workshop.

Opening lecture
Ute Kaiser (University Ulm) gave the opening presentation about the current state of the SALVE project and pointed to the tasks to be solved before the SALVE microscope will be handed over to Ulm University. Instrumentation-related tasks are the assembly of the instrument which was already shipped by FEI from Eindhoven to CEOS in Heidelberg in June, 2015. The team is at present waiting for the message "Beam down" with corrector! At the beginning of next year, the required specifications have to be demonstrated in the acceptance test. Since it was already measured at FEI in Eindhoven that the image spread of the base instrument is below the required limit at all accelerating voltages, the total image spread of corrector + base instrument should be sufficiently small to enable the predicted resolution of the final SALVE microscope. The technical task from the side of Ulm University is to construct the new SALVE building. The start will be in September this year and the end in late 2016. From spring 2016 until the end of 2016, the microscope will be operated by University of Ulm at the CEOS Company.

Important open application-related tasks are further understanding of radiation damage at the low acceleration voltages (20 - 80 kV), the cleanliness of sample preparation, and imaging of molecules on graphene. Ute Kaiser showed also the results of the evaluation of publications published in the field of aberration-corrected TEM, with respect to impact factors. The proportion of higher-impact publications (>10) in the low-voltage TEM field exceeds this proportion in the medium-voltage field by as much as a factor of 3. The evaluation showed that the SALVE team has the second highest output of high-impact publications in the low-voltage field worldwide (the highest output is produced by the University of Oxford, UK, for the trend graphs see the start page of the SALVE website). Ute Kaiser reported also on the recent collaboration between the two research projects dedicated to the development of low-voltage HRTEM, the SALVE project and the Triple C project in Japan.

For further information on the current state and future milestones of the SALVE III project see the news-article "Beam Down in the SALVE-III/TITAN Microscope" on the SALVE website.

Lectures in section instrumentation

Heiko Müller (CEOS) reported on the technical changes of SALVE+[1] C_C/C_S -corrector which the company performed when changing from the Libra to the Titan platform and on the necessary interfacing between corrector and microscope with respect to mechanics, vacuum and magnetic parts which was performed in close collaboration between CEOS and FEI. Now after delivery of the column in June 2015, the C_C/C_S corrector is being connected optically and mechanically to the column. Peter Tiemeijer (FEI) reported on the functionality of the FEI monochromator, which is part of the SALVE instrument. It is in best operation for HR-TEM. Furthermore, the company FEI presently develops a fully auto-alignment software for their monochromator which is necessary to produce the requested high brightness. He discussed in addition non-linear imaging in TEM which uses interference between both outer parts of lenses, similar to tilted-beam illumination and similar to STEM. Criteria how to distinguish between linear imaging and double scattering and non-linear imaging were demonstrated (the latter does not give frequency doubling). These non-linear contributions to the image can be minimized by focal series reconstruction. Moreover, he demonstrated the limits to resolve a large range of spatial frequencies (large Gmax / Gmin) and that high brightness of the gun/monochromator is required and can be achieved with present instrumentation.

Lectures in section theory and methods

Zhongbo Lee (University Ulm) gave a presentation on calculations performed in strong collaboration with Harald Rose of phase contrast achieved in aberration-corrected STEM equipped with a sectioning detector, which is based on earlier publications from Harald Rose[2]. Atoms behave like thin lenses characterized by negative and positive Cs and positive Δf. By mimicking the Fresnel phase plate with the aberration corrector, as well as collecting signal on a well-designed segmented detector, a magnified phase contrast image of the sample can be obtained.

Martin Linck (CEOS) explained the practical requirements which need to be fulfilled before phase contrast in STEM can be realized with a segmented ring detector. The calculations by Harald Rose showed that the correction of spherical aberrations higher than third order results in extremely large illumination angles. Martin Linck showed that without C_C-correction the unavoidable focus spread limits the image contrast in this mode severely. Even if the chromatic aberration is eliminated the present fine-tuning accuracy and precision are insufficient which might be avoided by employing another detector geometry, for example that of a pixel detector.

Christian Wacker (University Heidelberg) reported on further advances in understanding of image formation in the electron microscopes when considering backscattering, which he treated as an Eigenvalue problem. He reduced elastic scattering to a problem in linear algebra and demonstrated feasibility of his highly optimized numerical algorithms. He pointed out that his procedure offers the possibility for obtaining a unified description of image formation in SEM and (S) TEM. He showed that for thin objects the effect of backscattered electrons is rather tiny and argued that the effect of inelastic scattering should be more important which will be treated in due course.

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[1] Plus stands for the improved C_C/C_S corrector after optimizing the ray paths to reduce the image spread caused by Johnson noise (see Uhlemann, S., Müller, H., Hartel, P., Zach, J., & Haider, M. (2013) Thermal Magnetic Field Noise Limits Resolution in Transmission Electron Microscopy. *Physical review letters* **111**: 046101.).

[2] Rose, H. (1979) Phase Contrast in Scanning Transmission Electron Microscopy. *Optik* **39**: 416-436. and Hammel, M. & Rose, H. (1995) Optimum rotationally symmetric detector configurations for phase contrast imaging in scanning transmission electron microscopy. *Ultramicroscopy* **58**: 403-415.

Tatiana Gorelik (University Mainz) gave an overview on electron diffraction and pointed to the strength of this method compared to HRTEM when single-atom positions need to be determined in the case of radiation sensitive materials. She showed the comparison of different Cu-phthalocyanin layers in diffraction and HRTEM obtained in cooperation with Ute Kaiser (University Ulm).

Lectures in the section radiation damage and applications

Anita Ladenburger (University Ulm) gave an overview of different aspects of radiation damage observed during the first attempts to image biological objects on graphene. Results were reported for the Tobacco Mosaic Virus (TMV).

Johannes Biskupek (UniversityUlm) pointed to the general problem when imaging H-containing molecules, which can be reduced by exchange of H by D (deuterium). He also reported on experiments performed with Kazu Suenaga (AIST Tsukuba) for determining the elemental composition of clusters and the study of radiation damage to molecules on metal clusters and molecules inside CNTs at low voltages. The experiments have been performed on the 3C#1 instrument. Moreover, he showed the first results which Kazu Suenaga achieved on the new low-voltage microscope 3C#2, in detail of the 3rd order graphene reflection in TEM at 60 kV and the real space image of the graphene lattice at 15 kV.

Tibor Lehnert (University Ulm) gave an overview of the joined experiments with Rafal Dunin-Borkowski (Ernst Ruska Centre) and his team from the Ernst-Ruska Centre on radiation damage obtained with the PICO instrument operating at 50 kV. The results of the experiments with graphene performed by Lothar Houben (Ernst Ruska Centre) can be fully explained using the image calculations performed by Zhongbo Lee with respect to the contrast evaluation. However, the results with respect to the damage cross section for graphene/MoS_2/graphene performed on the PICO instrument could not be fully understood and necessitate further theoretical evaluation and experiments. Tibor Lehnert also showed results on imaging ionic liquids between graphene sheets in order to understand and find the formation conditions of square ice, and repeat the earlier experiments.

Felix Börrnert (Technical University Dresden) discussed his experiments and results obtained towards graphene devices in the TEM. Graphene has remarkable properties, as for instance extremely high charge carrier mobility and surface to volume ratio which make it interesting for sensors but also for many other applications [1].

Janis Köster reported on the influence of cooling on electron beam damage in the TEM. He discussed several new ideas in order to reduce the temperature during TEM imaging as well as its beneficial consequences for the reduction of radiation damage.

Thilo Zoberbier (University Ulm, Fig. 1) presented his results on catalysis with metals inside SWNTs, leading to a new view on the periodic table of elements on the atomic scale, which he conducted in tight cooperation with Andrey Khlobystov (University Nottingham). The dynamics show a previously hidden symmetry, which allows classifying this system and extracting the most important physical properties without the need to know all reactants in detail.

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[1] Ferrari, A. C., Bonaccorso, F., Fal'Ko, V., Novoselov, K. S., Roche, S., Bøggild, P., ... & Van Wees, B. J. (2015). Science and technology roadmap for graphene, related two-dimensional crystals, and hybrid systems. *Nanoscale* 7: 4598-4810.

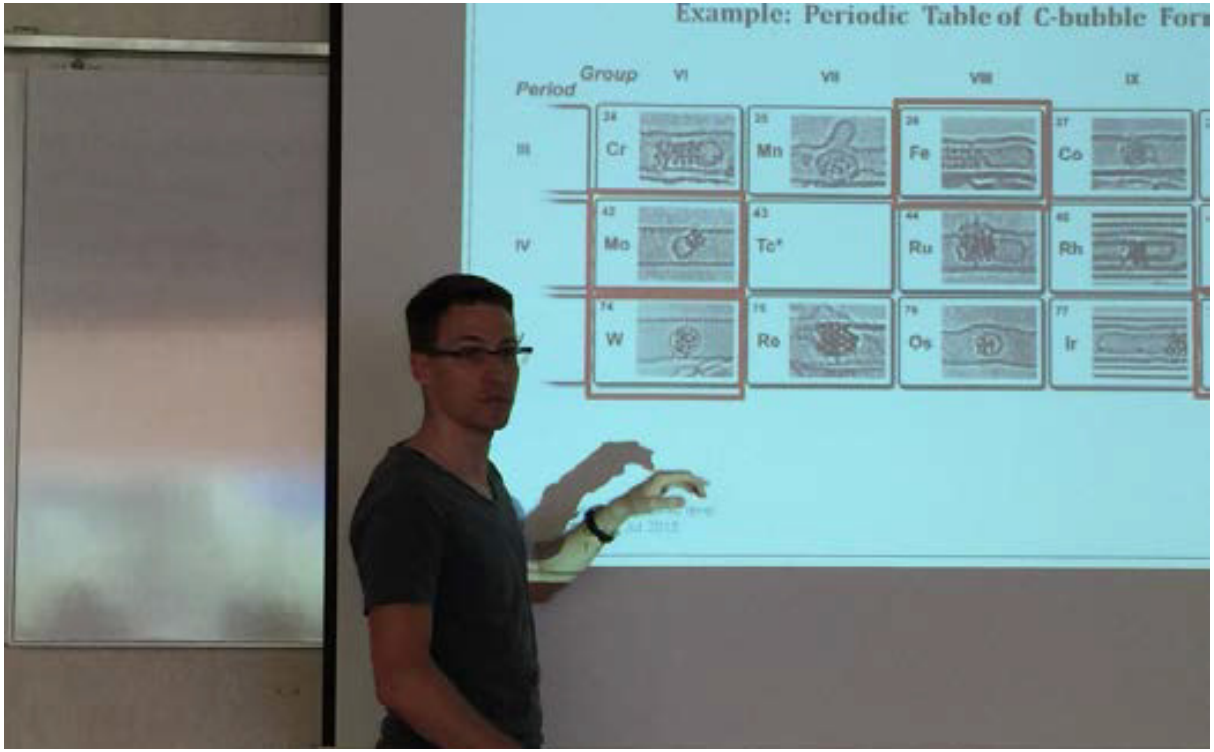

Fig. 1: Thilo Zoberbier discussing his periodic table of elements in the atomic scale for 13 metal clusters inside CNT.

Micheal Kinyanjui presented his current work on charged density waves. This new topic combines results on super conductors and Li-ion battery materials, which is advantageous because it allows for ultra high capacity energy storage.

Ute Golla-Schindler discussed the results of her studies on battery materials using combined SEM and TEM observation. These materials find applications in energy physics, but also in fundamental studies of intercalation.

Anita Ladenburger, Pia Knyrim and Robert Leiter reported in their evening lecture about the TEM Summer School in Antwerp which they attended just before the SALVE workshop. Their report confirms the methodological diversity and physical span of transmission electron microscopy.

The 5th SALVE workshop documented the wide range of the tasks within the SALVE III project: further understanding and reduction of radiation damage, development of specimen preparation, instrument development, better understanding of imaging theory for HR-TEM and spectroscopy in order to image beam-sensitive objects with higher resolution than currently possible.

WORKSHOP PROGRAM

Tuesday, June 29

15:00 - 15:30 Welcome and state of the SALVE Project
- U. A. Kaiser

15:30 - 16:00 Installation of SALVE III at Heidelberg
- H. Müller

16:00 - 16:30 Evaluation of 50 kV PICO experiments
- T. Lehnert, Z. Lee, J. Biskupek

16:30 - 17:00 Experiments on encapsulated CNT in STEM @ Tsukuba
- J. Biskupek

17:00 - 17:30 First attempts to image biological objects
- A. Ladenburger

17:30 - 18:00 Preparation and observation of 2D-objects between graphene
- T. Lehnert

20:00 - 21:00 Discussion: Radiation damage at low voltage

Wednesday, June 30

8:30 - 9:00 Some work on HR-TEM, non-linear imaging, brightness, monochromator, and auto-tuning
– P. Tiemeijer

9:00 - 9:30 STEM phase contrast (1)
- Z. Lee

9:30 - 10:00 STEM phase contrast (2)
- M. Linck

10:15 - 10:45 Backscattering as an Eigenvalue problem
- C. Wacker

10:45 - 11:30 Electron diffraction
- T. Gorelik

12:00 - 18:00 Miniworkshop

20:00 - 21:00 After-dinner-talk

Thursday, July 1

8:30 - 9:00 Towards graphene devices in TEM
- F. Börrnert

9:00 - 9:30 Influence of cooling on electron beam damage in MoS_2
- J. Köster

9:30 - 10:00 Catalysis with metals inside CNTs on the atomic level
- T. Zoberbier

10:00 - 10:30 Current status: Charge density waves observed in 2D materials
- M. Kinyanjui

10:30 - 11:00 Current status: Battery materials
- U. Golla-Schindler

11:00 - 11:30 Final discussion and summary

To further ask questions is the best way for getting deeper and deeper understanding and improved knowledge! On the next, the 6th, SALVE workshop we will present proof of our theoretical and experimental evaluation by showing relevant results obtained with the completed SALVE-TEM.

Group photo of the 5th SALVE Workshop in Hirschegg, Kleinwalsertal

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