Universal scanning electron microscope as a multi-nano-analytical tool

PROJECT FINAL REPORT

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Name of the scientific representative of the project's co-ordinator (Title and Organisation):

Jaroslav JIRUŠE, PhD.

TESCAN Brno, s.r.o. (Czech Republic)

jaroslav.jiruse@tescan.cz

+420 547 130 468

Project website address: http://univsem.eu

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1. An executive summary

A main aim of the UnivSEM project was to introduce the new approach in in-situ analysis, enabling various combinations of analytical add-ons and complementing the desired structural information piece by piece. In one multimodal instrument based on Scanning Electron Microscope (SEM) and Focused Ion Beam (FIB) platform, the Confocal Raman Spectroscopy, Time of Flight analysis and Scanning Probe microscopy are integrated. The capability is further widened by the means of 3D tomography.

Even though all the particular techniques bring the unique information about the sample of interest, combining these all in one instrument brings several synergic effects. The main benefit can taken from the system modularity is the possibility to analyse one single place in the sample without the necessity to move the sample outside SEM chamber.

During the three years of project duration, three UnivSEM prototypes were introduced. The very first microscope with the integrated Confocal Raman Spectrometer (CRM) and the novel immersion optics in SEM column was introduced in 2014 on Analytica in Munich. The fully integrated system of unique Ga FIB-SEM instrument was presented in September 2014 on International Microscopy Congress in Prague. The realization of the third prototype was enabled by the joined effort of the TESCAN and its daughter company – Orsay Physics (Orsay costs not charged into the project). In the first stage of the project, the integration of unique Xe Plasma FIB into TESCAN microscopes with the immersion lens was designed and the first mechanical drawings were released. The verification of these documents was not originally involved in the planned project activities, but were added in the amendment during the second year. Additional effort was invested and the novel Xe Plasma FIB-SEM with multimodal chamber accommodating various add-ons and the immersion lens was successfully assembled and tested in 2014 and 2015. Due to high rate of modularity, the performance of both instruments can be easily compared, which was demonstrated on the example of TOF-SIMS spectrometry.

There are several key aspects, leading to the success of the UnivSEM project. Firstly, there is the strong and well-established consortium with balanced number of participants from the industrial and non-profit sector, coordinated by the high-tech and innovative SME (LE) – TESCAN. Secondly, all the add-ons developed by the industrial beneficiaries, were integrated to the same platform, by the main integrator and project coordinator TESCAN. Such win – win approach is highly preferred by the end customer, who can easily ensure the future service contract. Thirdly, the academic partners provided highly relevant scientific input. These were responsible for the user feedback, application development and dissemination. During the project, we established also the cooperation with the metrology and validation activities on European level.

2. A summary description of project context and objectives

The UnivSEM project scope was fully aligned with TESCAN R&D priorities. The project was strictly product oriented, bringing the new capability into nanotechnology. Nowadays, two main trends might be observed in the nanotechnology. Firstly, nanotechnology devices producers are aiming to the best resolution possible to be able to discover the surface on the molecular level. The bottleneck of this approach are the instrumentation limitations. The second trend observed is the requirement of increasing functionality of the scientific instrumentation.

The first multimodal scanning electron microscope chamber was designed within the 7th Framework Programme project called FIBLYS (www.fiblys.eu). The well established project consortium was further widened for industrial partners – Witec, the specialist on Confocal Raman Spectrometry and Brno University of Technology with the aim to increase the system capability and function. Following goals were stated at the beginning of the project

- a vision capability
- a chemical analysis capability
- structural characterization
- nondestructive optical capability
- manipulation and structural capability

All the system functionality is integrated into one platform and by one integrator TESCAN. The project was divided into eleven work packages, including also the management and dissemination one. Here, the brief overview of the project outline is introduced, the scientific outcomes are discussed in chapter 3.

In the first work package, the vacuum chamber was designed with respect to the geometrical interplay of all methods. It is worth to note, that we do not expect the end – customer to buy the fully equipped system, but according to all the marketing studies we did, the various combinations of add-ons available are required. As the output of the work package, the mechanical drawings of all the add-ons are placed in the most favorable position for their performance. The leading partner for this activity was the project coordinator – TESCAN with the contribution of all industrial partners.

The second work package was devoted to the focused ion beam technique integration, again with main contribution of TESCAN R&D engineers. The two types of ion source integration were designed combining the novel immersion optics introduced within the project with the ion columns supplied by the traditional TESCAN partner – Orsay Physics. All the drawings led to the production of the UnivSEM prototypes.

The first add-on integration was done in work package 3, where the technique of Time Of Flight analysis is introduced and utilized to be part of the complex system. The R&D activities in the work package were lead by Swiss partners, the industrial company TOFWERK with the contribution of EMPA in the performance testing. The developed analyzer was further sent to TESCAN premises in Brno, where it was integrated to the newly manufactured chamber. These activities are closely related to the work package 4, where the influence of gas injected in the system were discovered and several approaches were suggested, leading to the increasing yields of secondary ions, both negative and positive.

The modularity of the final instrument was widened in work package 5, mainly by contribution of German SME – SPECS, who is focused on surface analysis by means of scanning probe microscopy. In the first part of the project, the Stand-alone SPM was introduced, which was consequently integrated into the SEM chamber.

The missing capability in optical microscopy was fulfilled by the integration of the confocal Raman Microscope (work package 6). Here, the leading partners Witec and TESCAN were aware of the current intellectual property related to the point of integration. Two various conformations of the set up were introduced and both covered by the shared patent application. Two prototypes of Raman microscope in the chamber were produced, the first one placed in Witec, the second one as the part of final UnivSEM prototype.

For the overall system functionality, the most important issue is the complex system integration.

Figure 1. The UnivSEM system (trade name GAIA)

The integration, including the software control was performed in TESCAN in cooperation with all the industrial partners. The crucial point of the integration was the highly accurate design of single add-ons within WP1 to WP6. Due to the well managed partners cooperation, the integration was successful and the prototype was introduced to the general public.

The UnivSEM outcome as the contribution to the metrology and standardization activities was presented in work package 8. The high level of correlation enables the validation of one technique among the other one. The standardization and metrology seems to be a very promising field for future prototype usage.

Even though the system developed in the project is world-wide unique, it is necessary to introduce it to the end customers. For that purpose, the applications (Work package 9) and Dissemination (Work package 10) activities are necessary.

Last, but not least, the carefully taken management activities were presented in the project. The regular project meetings of all partners and the meetings organized by work package leaders ensured the smooth project progress.

The UnivSEM project objectives were stated as follows:

1. To design the vacuum chamber for complex and modal nanoscale analysis.
2. To integrate both Ga and Xe plasma focused ion beam
3. To develop the orthogonal TOF-SIMS spectrometer with the improved sensitivity up to 30x and lateral resolution (3x)
4. Allow 3D tomography
5. Integration of scanning probe methods
6. Integration of optical methods
7. Complex software integration

All the defined goals were successful in the given delivery dates and the UnivSEM prototype is ready to be commercialized.

3. Description of the main S&T results/foregrounds:

3.1. Time-of-flight (TOF-SIMS) - elemental analysis

TOF stands for Time-of-Flight and in this context refers to Time-of-Flight mass spectrometry. Mass spectrometry is a technique where the mass-to-charge ratio of ions is measured in order to identify them. TOF is a particular implementation of this technique with advantages including the rapid acquisition of data (up to one hundred thousand mass spectra for second) and the simultaneous detection of all masses – both essential for rapid imaging of nanostructures. SIMS stands for secondary ion mass spectrometry and is a classical near-surface analysis and imaging technique with low detection limits, high surface sensitivity and high spatial resolution for a variety of materials. It is also one of the few analytical techniques that can differentiate between different isotopes of the same element, thus allowing tracer studies. TOF-SIMS is SIMS using a TOF mass analyser.

The objective of this work package was to improve an existing TOF-SIMS accessory for FIB and to adapt it to the instrument developed within the UnivSEM project. The improvements can be divided into improvements of the performance of the instrument and adaptations to make the instrument interfere less with other instruments within the FIB chamber thus fitting better in the multimodal UnivSEM instrument.

The work package also included the development of new acquisition hardware and software. In addition, a residual gas analyser has been developed that allows high mass resolution and rapid measurementsof the gas composition in the FIB chamber using an electron impact source and the mass analyser of the TOF-SIMS add-on.

The efficiency of the extraction optics is very tightly linked to the distance between the extraction optics and the sample. However, because of the multiple measurement techniques available with the UnivSEM instrument, there is very little space close to the sample. For this reason, a slide has been developed allowing moving the TOF-SIMS accessory in and out of the chamber. The strict space requirements prevented using standard parts for this so a custom solution has been developed and tested.

To complement the slide, a new ion optic design has been developed that has a superior transmission efficiency compared to the previous systems. The new setup has also been equipped with positional adjustment screws that allow lateral positioning of the ion optics. This ensures that optimal performance of the system can be achieved without excessively demanding engineering tolerances for the microscope flange.

The TOF-SIMS accessory has also been equipped with a gate valve. The gate valve allows the TOF-SIMS mass analyser to be kept under vacuum even when the FIB chamber is vented. It lowers the time to pump down the system to sufficient vacuum levels since only the FIB-SEM chamber has to be vented. It also helps protect the TOF-SIMS as it contains sensitive detectors that cannot be exposed to air for extended periods.

Another adaptation of the TOF-SIMS accessory is the inclusion of a metal shield covering the ion optics. The purpose of the shield is to lower the electrostatic interference between the TOF-SIMS and other techniques within the UnivSEM instrument as well as to protect the TOF-SIMS accessory from accidental short circuits and even mechanical damage.

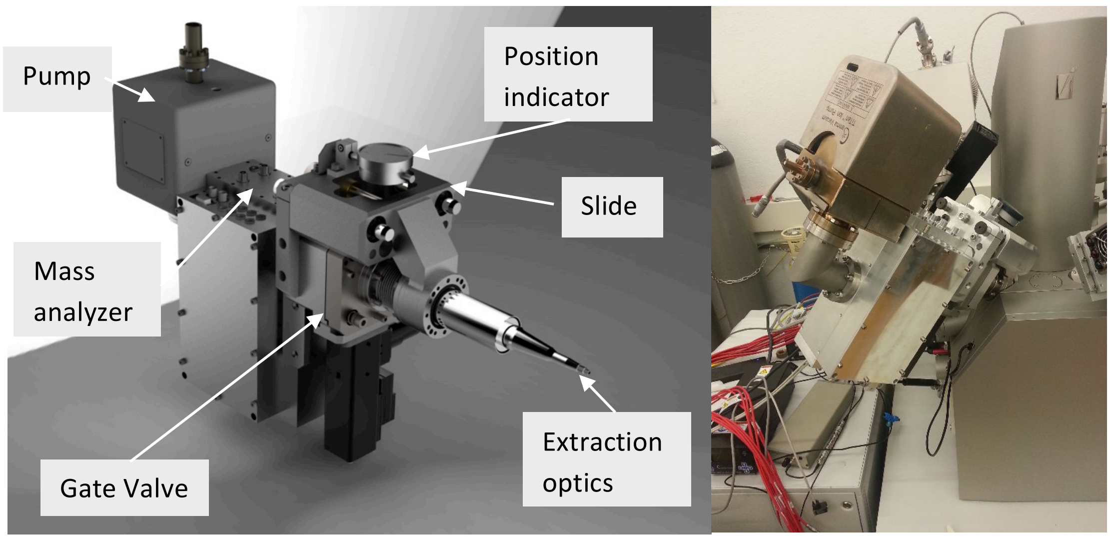

Figure 2: Left: CAD rendered image of the full TOF-SIMS accessory. Right: Photo of the TOF-SIMS accessory installed on a FIB chamber at EMPA

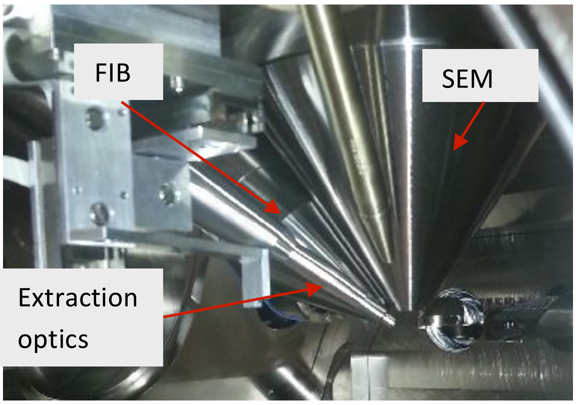

Figure 3: Photograph of the inside of the FIB chamber showing the SEM, FIB and the TOF-SIMS Extraction optics

On the electronics side, a new acquisition card has been implemented that allows for faster measurements.

As for any complex measurement technique, TOF-SIMS has to be backed by sophisticated software. Combined acquisition and visualization software has been developed for this purpose. The software allows relatively easy acquisition of data and basic imaging and chemical identification. In addition special software has been developed allowing automatic optimization of the many electrical potentials in the ion optics of the instrument to achieve optimal performance. This is faster and more reproducible than attempting manual optimization.

As with any vacuum system, the vacuum in the FIB chamber is not perfect. The residual gas remaining in the chamber after pumping can have an impact on measurements performed (e.g. SIMS measurements, FEBID/FIBID deposition and etching). The composition of the gas in the vacuum chamber can be analysed using a residual gas analyser (RGA), but such an instrument is relatively expensive (>20 kEUR). By installing a so called electron impact source it is possible to ionize the rest gas of the FIB-SIMS chamber. Combining this with the TOF-SIMS analyser gives a residual gas analyser at low additional cost and with much better mass resolving power than commercial RGA instruments that use quadrupole mass filters. This has been implemented and tested.

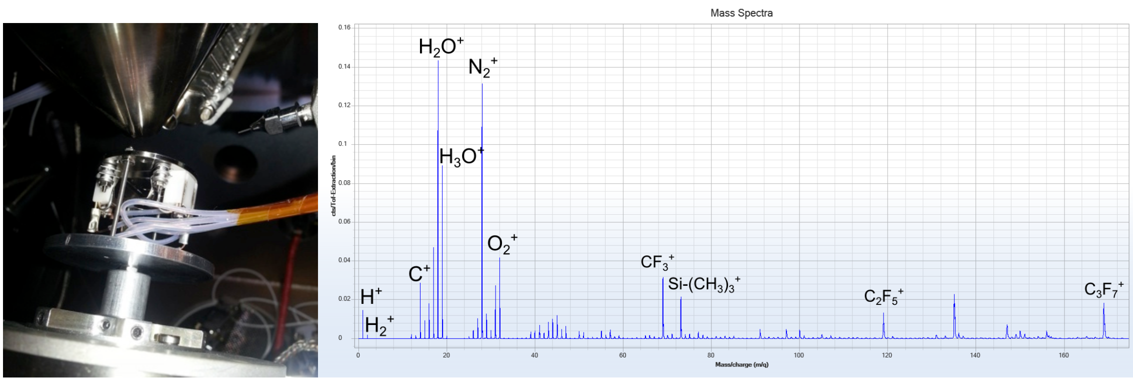

Figure 4: (left) Electron impact source installed at Empa and a mass spectrum showing some molecules detected in the residual gas

3.2. Control of surface chemistry for enhanced sensitivity

The main aim of this work was to improve the sensitivity of the UnivSEM secondary ion mass spectrometer (SIMS), which enables the imaging (in three dimensions) of the distribution of chemical elements in a sample. Features as small as 40 nm, or as thin as 5 nm can be seen. The advantage of the FIB-SIMS approach adopted by the UnivSEM project is that it provides for a very versatile chemical imaging platform that can incorporate many instrumental techniques and accommodate large and irregularly shaped samples. The disadvantage of this approach is that the sensitivity of the SIMS instrument suffers. In order to partially compensate for this, we can alter the surface chemistry of the sample in such a way as to increase the SIMS signals. We can do this either by allowing oxygen into the vacuum chamber, or by coating the sample continuously with caesium as we measure. The former approach is good for improving signals from such elements as aluminium, silicon, boron (electropositive elements) and the latter approach is good for improving signals from elements such as chlorine, arsenic, phosphorous, gold, carbon and hydrogen.

Secondary goals were to thoroughly test the improved SIMS system developed by the project, and to explore its use as a residual gas analyser for monitoring the quality of the vacuum system and for finding the cause of leaks.

The use of oxygen flooding was confirmed to give at least a 10x increase in sensitivity for several elements and matrices, including transition metals from an alloy. (This material was developed and analysed by Empa because of the possibility of using it to make cheaper and better electric motors). The use of caesium flooding was also shown to give almost an order of magnitude increase in sensitivity. This is the first time that this has been shown in a SIMS instrument operating under 'high vacuum' conditions, instead of the more typical 'ultra high' vacuum in which the system pressure is 100 to 1000 times lower.

The improved ion optics and incorporation of a slide to adjust the working position of the mass spectrometer gave increased sensitivity compared to the start of the project. The incorporation of a valve between the mass analyser and the microscope chamber, and the use of the slide to keep the mass spectrometer out of the way of bulky samples and other instruments greatly contributed to ease of use and faster pump-down times and both are regarded as essential by Empa for general commercial success.

We successfully showed that the addition of a simple electron impact ion source to the microscope chamber (situated on the sample stage) allowed the UnivSEM mass analyser (of the SIMS component) to be a residual gas analyser (RGA) with much better mass resolving power and much faster measurement than the usually used quadrupole RGA. This instrument confirmed the already noted presence of a fluorocarbon background in the chamber at Empa, which is believed to arise from lubricants used in the microscope stage.

Taken together, the surface chemistry modification and hardware improvements gave an improvement in the SIMS signal during the project by a factor of 75. With this improved signal, we could still show a lateral resolution of better than 40 nm with a mass resolving power of over 3000 on the Empa prototype.

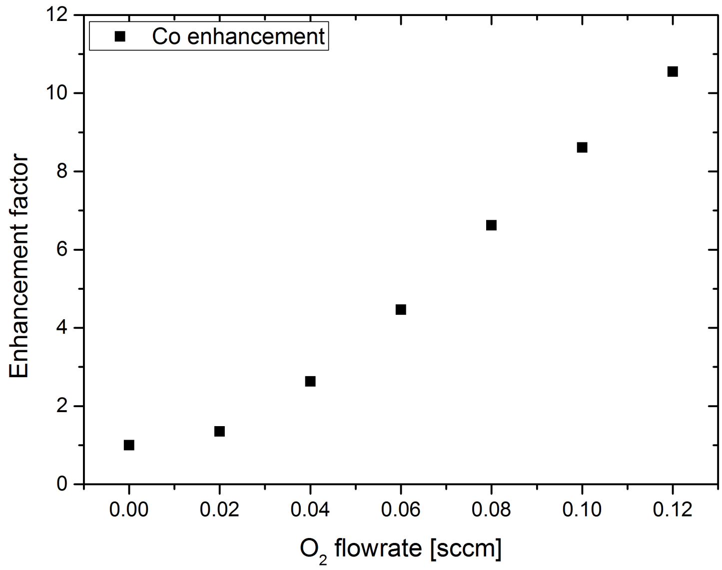

Figure 5: More than x10 increase of SIMS signal when oxygen is bled into FIB chamber. Measurement conditions: 60 pA, 20 keV Ga+, 40x40 mm; sample is a CoNiFe alloy. Data shown are for 59Co+ secondary ions, and have been normalized to zero oxygen flow.

3.3. Integration of optical methods in Scanning Electron Microscope – molecular analysis

As a result of the UnivSEM project, a confocal Raman microscope for operation in the vacuum chamber of a scanning electron microscope was developed and successfully built. The idea behind the project was to be able to correlate high-resolution SEM images with the structural and chemical analysis technique Raman imaging. The system is called RISE (Raman Imaging and Scanning Electron) microscopy and was first introduced to the public at the Analytica fare in Munich, Germany in April 2014.

Prior to this project, the existing Raman/SEM combination systems only allowed the acquisition of a single Raman spectrum from a selected point on the sample and not the acquisition of a complete image. This was a big disadvantage, because the alignment between SEM and optical microscope is very critical and subject to thermal and mechanical drift, as well as drift through sample charging in the SEM beam. Prior to this project, the existing Raman/SEM combination systems used an off-axis parabolic mirror to excite the sample and collect the Raman scattered light. These systems are not confocal and off axis parabolic mirrors are very sensitive to misalignment and have a limited field of view.

Due to the use of a high quality, high NA, vacuum compatible microscope objective (100x, NA=0,75) with large working distance (4mm), the resolution of the Raman microscope could be improved by a factor of 5-10 times compared to existing Raman/SEM combinations. At the same time, a very high sensitivity was achieved due to the high collection efficiency and transmission of the microscope objective used. Additionally, the system is confocal and therefore capable of 3D imaging with a depth resolution of below 2µm. The use of a high-resolution piezo-scanner with 250x250x250µm scan range allows ultra precise measurements with a scaling error well below the diffraction limit of the optical beam.

In the project, a software package was developed that allows the seamless overlay of SEM and confocal Raman images to benefit from both, the high spatial resolution of the SEM and the chemical information of the Raman microscope.

Figure 6: The newly developed RISE microscope integrates a SEM and a confocal Raman imaging microscope within a single instrument.

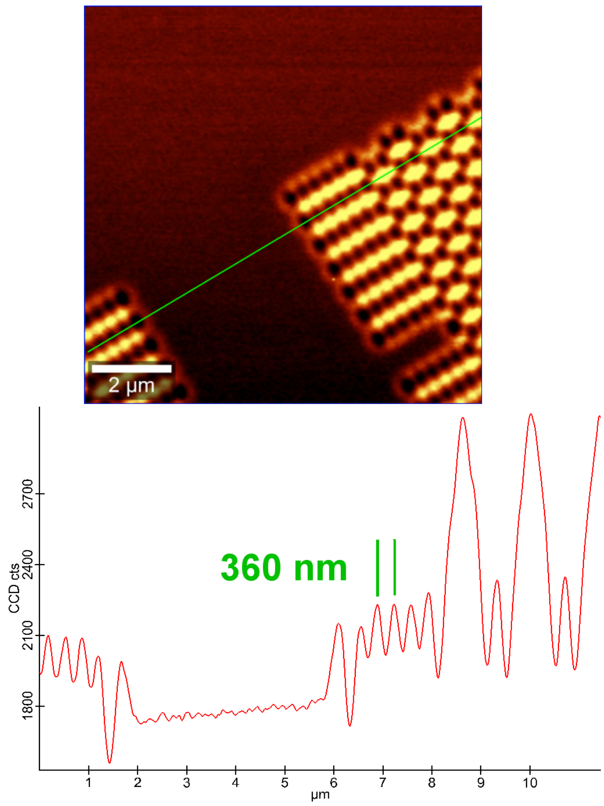

Figure 7 : Raman image (above) of a hole structure in Si using 532nm excitation. The image shows the intensity of the Si signal at 520cm^{-1} in which the holes appear dark. The cross section on the right shows the intensity along the green line. The holes with a spacing of approximately 360nm are clearly resolved.

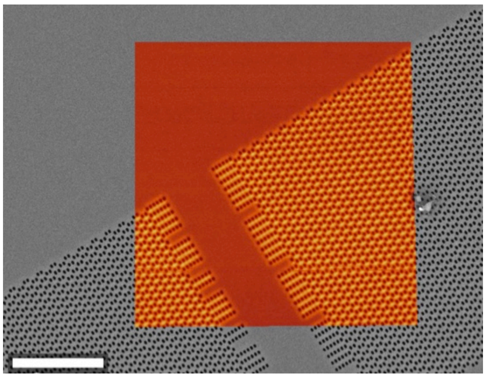

Figure 8: Overlay of SEM (gray) and Raman image (intensity distribution of the Si signal, orange)

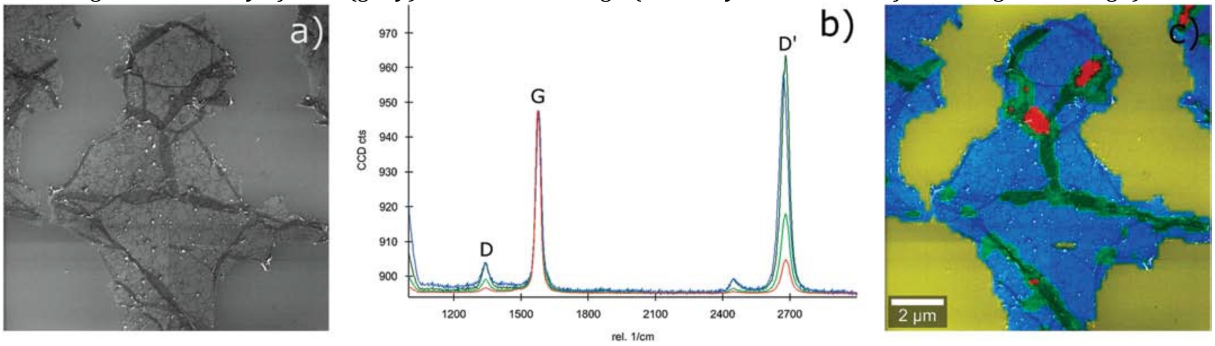

Figure 9: RISE microscopy of a CVD graphene film in high vacuum. (a) Secondary electron SEM image of the graphene film revealing defects in the surface structure. (b) Main Raman spectra identified by differences in the peak width and intensity. (c) Corresponding color-coded, confocal Raman image. The colors show the graphene layers and wrinkles (blue indicates a single layer, whereas green and red areas are multi-layer). The silicon substrate is shown in yellow.

3.4. Scanning probe methods in SEM – approach to precise topography

The aim of this work was to complement the UnivSEM tool with a scanning probe microscope (SPM). The project partners designed, produced, and tested two SPMs with < 0.1 nm z-resolution, 10-fold increased scan speed, 25-fold increased field of view, and 3 mm working distance. In addition, the SPM control software was prepared for integration of SPM with SEM and with TOF-SIMS. One of these two SPMs served as a development bench for testing the performance under ideal stand-alone conditions. The other SPM has been integrated into the UnivSEM demonstrator and served the purpose of testing and optimizing the challenges of the integration into the complete UnivSEM tool. The SPMs are completely designed and optimized for the specifications needed in the UnivSEM tool. The two microscopes were designed, built and tested by the middle of the project, such that enough time could be devoted to the integration phase.

Achievements:
The integration of a scanning probe microscope (SPM) into a scanning electron microscope (SEM) or a focused ion beam tool (FIB) is motivated by the fact that these techniques offer very complemental information to the user.

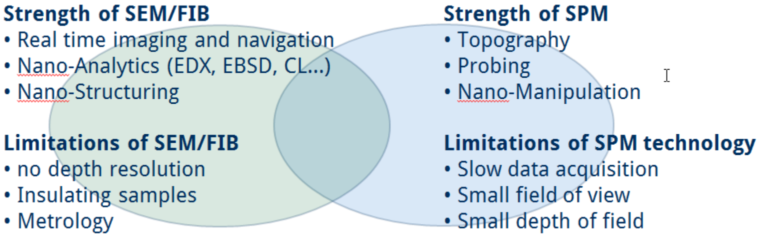

Figure 10: Complementarity of SEM/FIB and SPM

In order to combine the SEM technique with SPM, a list of specifications is required that makes the use of a standard commercial SPM impossible. These specifications include

- Vacuum compatibility: the SEM requires a SPM that is high-vacuum compatible
- Compactness: the free space inside the SEM chamber is very limited. The combination of standard SEM-tools plus the additional techniques in the UnivSEM tool pose strict limits on the geometry of the SPM. The main challenge here is that the half-space above the sample must be kept free of SPM-related parts.
- Precision: the SPM needs to be mounted on the SEM stage, which is not optimized for sub-nanometer stability measurements. On the other hand, length scales of below 0.1nm in depth and below 1nm laterally are needed as an enabling technology in this instrument.
- Speed: the imaging speed of the SPM must be high enough in order to make an intertwined measurement of SPM with other techniques possible.
- Field-of-view: The possible fields-of-view of the SPM and the other techniques in the UnivSEM should overlap as much as possible. This means that the field-of-view of the SPM has to be large as compared to standard SPMs.
- Integration: the user of the SEM should experience the interaction with the SPM as natural extension of the SEM workflow.

Those specifications lead to particular design choices of the instrument.

The SPM design is based on a tripod design as opposed to a traditional stacked design. This allowed us to have the space above the sample almost completely available for other techniques.

a) b)

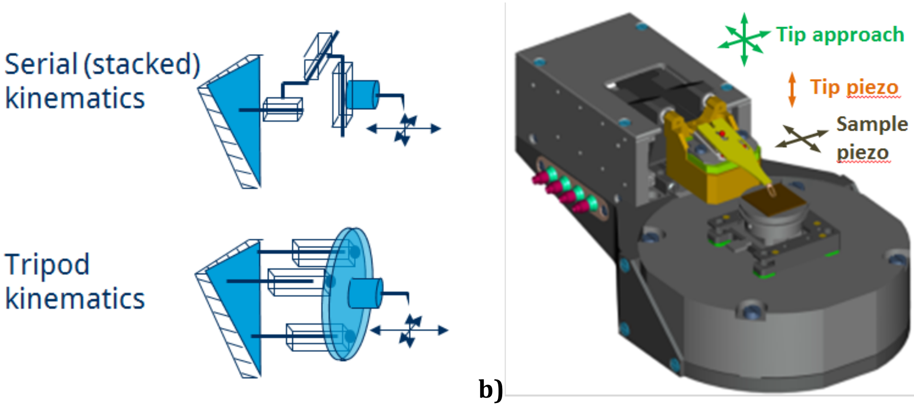

Figure 11: The tripod design allows for a very compact SPM (a). The final layout of the UnivSEM SPM. The round base houses the large area x-y-scanner that enables large field-of-views (b).

The SPM uses self-sensing force and current sensors called Akiyama-probe and Kolibri sensors. Self-sensing SPM-sensors don't require a laser above the sensor and thus make the open design possible. The Akiyama-probe sensor is a commercial sensor that has been adapted in its design for this instrument. The Kolibri sensor is a SPECS product that needed to be redesigned in order to be compatible with this SPM. The Kolibri sensor is a very fast sensor that allows for faster imaging speeds and thus helps the integration with the other techniques in the UnivSEM.

a) b)

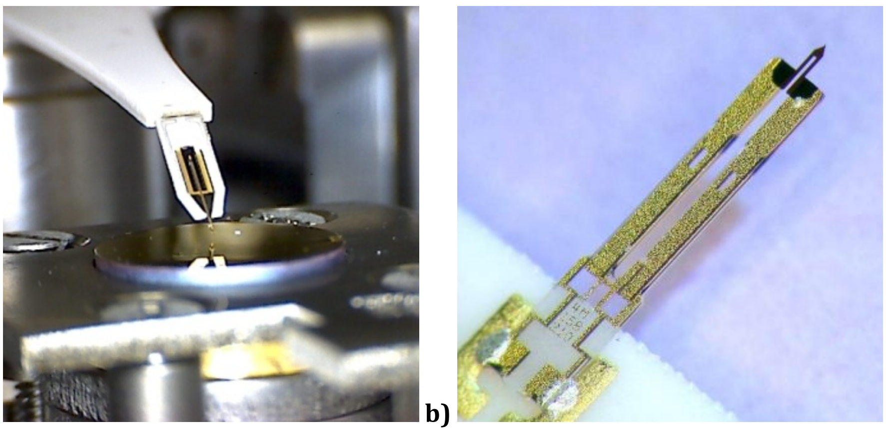

Figure 12: Kolibri sensor in the SPM, imaging a gold crystal (a). Akiyama-probe sensor (b)

The stability and resolution of the SPMs built within this work package is proven by measuring on particular test-samples like test grids, graphite crystals and gold crystals. The instrument fulfilled our stability specifications.

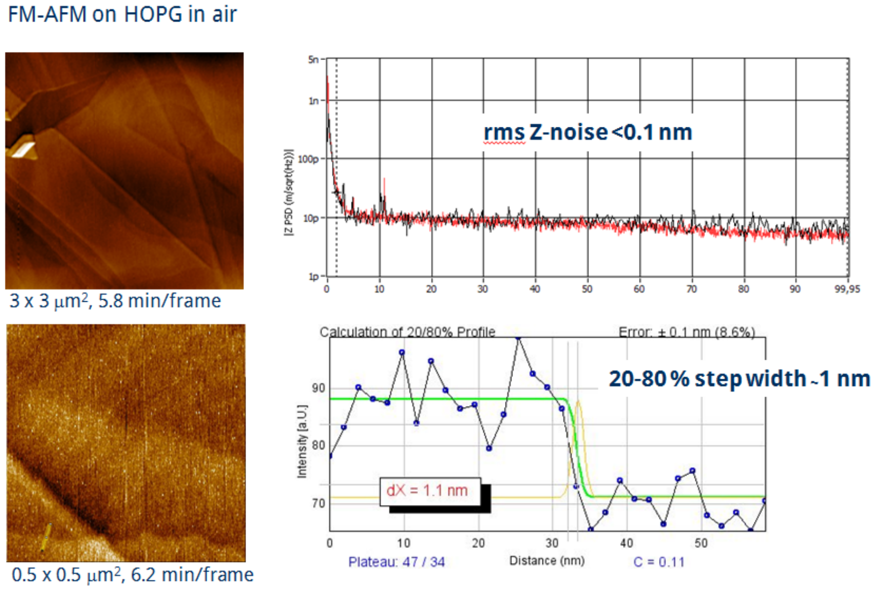

Figure 13: Z-stability measures on single crystalline graphite (HOPG). The right spectrum shows the stability at different frequencies and proves that the overall stability is below 0.1 nm ***(top)****. The lateral resolution is shown in a line profile over an atomic step. The lateral resolution is better than 1 nm* ***(bottom)****.*

The software-package for running the SPM is the standard-SPECS software package that is designed for specialized SPM users. In order to make the SPM part of the UnivSEM tool, the software needed significant changes and additions that enabled us to integrate it into the SEM control software. The underlying software structure that needed to be implemented was a universal remote control interface for the SPM. The SEM software uses this interface and presents the user of the SEM with a set of simple procedures that integrate the SPM into the SEM-work-flow.

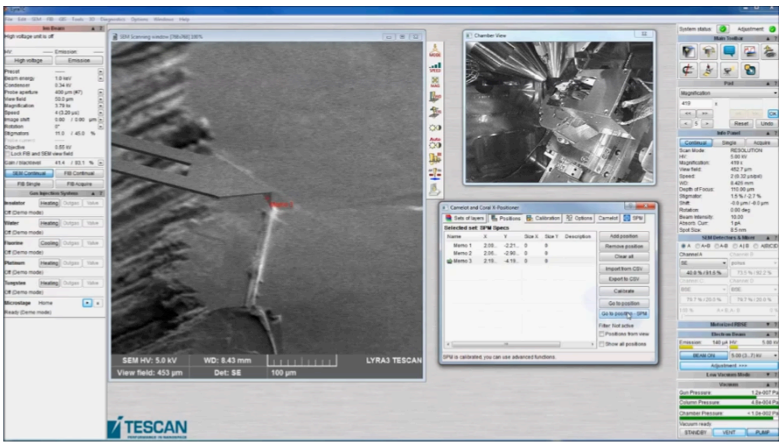

Figure 14: Screenshot of the SEM software that shows the integration of SPM-related features into the SEM software. Simple and repeated tasks, especially related with positioning the tip, can be performed directly from the SEM software without the need to work in the SPM software.

The integration of one of the two SPMs into the UnivSEM tool was an important milestone for starting to work on the multi-technique tool. The mechanical integration worked smoothly and the detailed integration work with the other techniques could start in time.

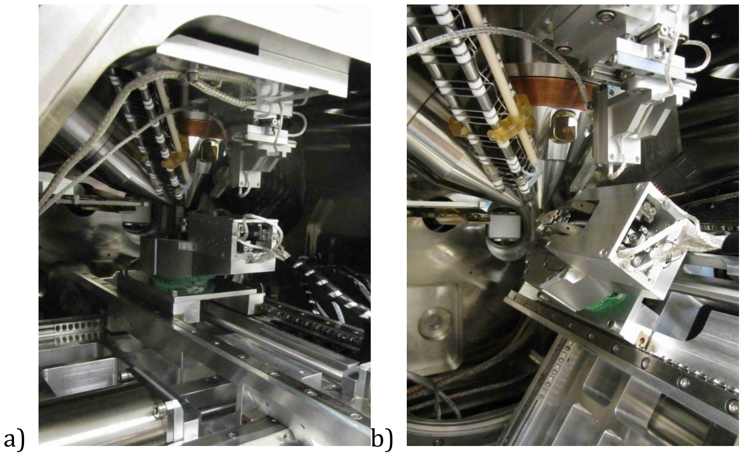

Figure 15: The SPM is mounted on the new manipulator in a position (a) towards the FEB column (vertical) and (b) towards the FIB column (left, tilted).

One task within this work package was to explore the potential of local electric field induced deposition (LEFID). In this technique, the tip of the SPM is brought close to the surface and a voltage is applied. At the same time a chemical precursor gas is introduced into the SEM chamber that is decomposed by the electric field such that a defined deposition of material under the tip is possible. In order to work with this technique, the SPM has to be positioned in the immediate vicinity of the gas injection system (GIS) of the SEM/FIB.

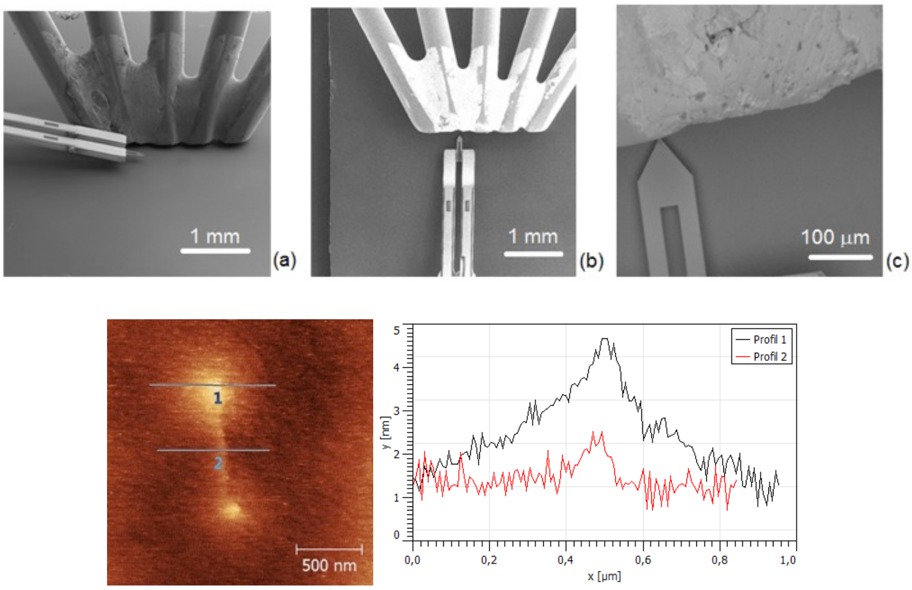

Figure 16: Sequence of images showing the positioning of the SPM tip as close as possible to the gas injection system (top). Deposited material after applying a defined voltage to the tip (bottom).

The applications of the combination of the two techniques are manifold, and we only show a few impressive examples here.

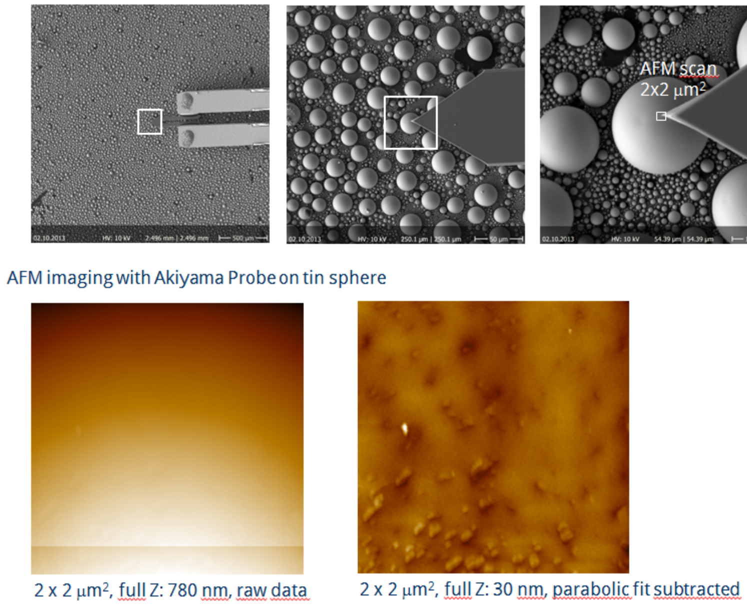

Figure 17: The SEM beam can be used to guide the SPM tip to areas that would have been impossible to access without the help of the SEM imaging capability.

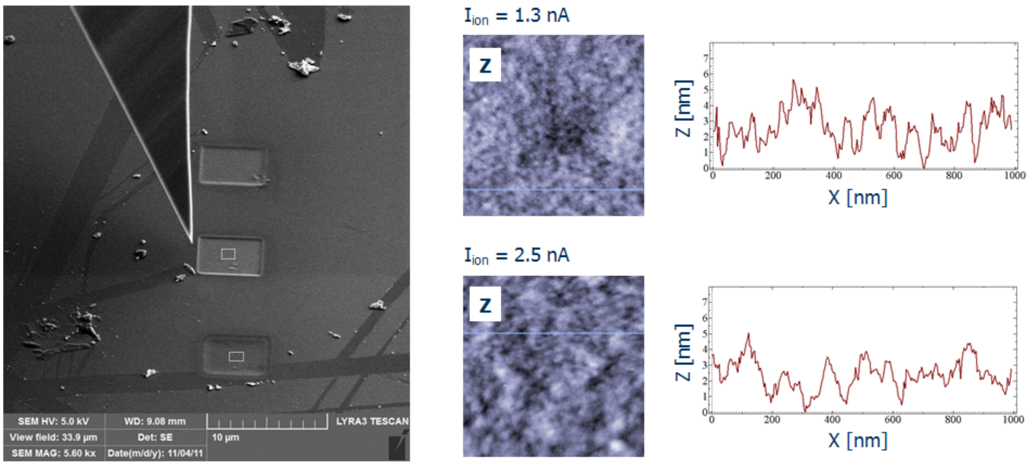

Figure 18: The AFM is used to measure the roughness at sub-nanometer scale at the bottom of a FIB-milled crater.

3.5. Perspective applications of the UnivSEM

The consortium aimed for a diverse demonstration of the analytical capabilities of the UnivSEM tool. Therefore, the academic partners MPL, EMPA and BUT utilized their extensive equipment and experience in the fields of photovoltaics, plasmonics & complex nanostructures to generate suitable sample systems for sophisticated benchmarking of the different analytical add-ons.

The UnivSEM products RISE, GAIA & XEIA enable the analysis of samples from nearly all research topics like materials science, geology, forensics, semiconductors, biology and more. The analytical add-ons are able to gather topographic, tomographic, chemical, compositional, crystallographic and molecular information. The correlative combination of such different measurements is one of the hottest topics in microscopy nowadays.

Here, we would like to present the results of the analysis of three different materials systems:

- Graphene on top of Gallium Nitride nanorod LEDs
- A silicon thin film solar cell
- An industrial planar GaN LED

Graphene on top of Gallium Nitride nanorod LEDs

Light emitting diodes (LEDs) made from gallium nitride (GaN) have recently jumped into the spotlight due to the Nobel Prize in Physics 2014 which was awarded to researchers from Japan. Energy efficient lighting is one of the most important topics in todays' semiconductor industry and has an immense impact on the real world. At the Max Planck Institute we are working on a new type of nanostructured GaN-based LEDs. As front contact to the diodes we apply another 'hot topic' & Nobel Prize winning material: graphene. This carbon-based two-dimensional ultrathin material has nearly perfect physical properties for the mentioned application: it is nearly transparent for light and it is a very good electrical conductor.
For the investigation of such a combination, the UnivSEM tools are ideally equipped with the best analytical tools. Especially, high-resolution Raman Spectroscopy is a technique for in-depth analysis of carbon based materials.

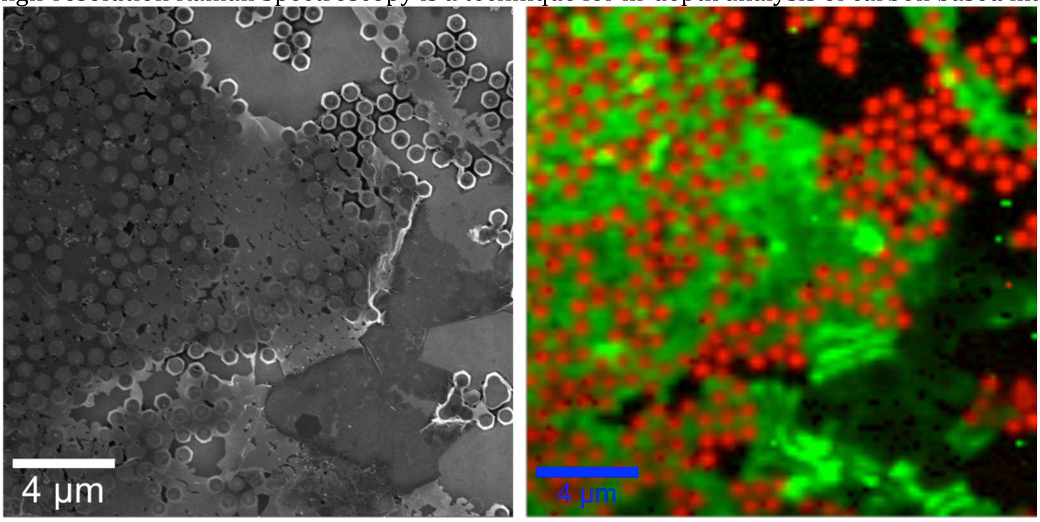

Figure 19: Left: SEM image of GaN nanorods, partially covered with a thin layer of semi-transparent graphene. Right: Raman spectroscopy measurement that shows the GaN in red and the graphene in green. Sample courtesy of Michael Latzel, MPL.

Figure 19 shows SEM and Raman measurements that were obtained at the same position, which is a powerful and important feature of the UnivSEM tool. Even though the graphene is only one atom thin, optical characterization is an easy and straightforward task. In combination with a highly-resolved SEM image, features and/or defects can be addressed to structural changes at the nanoscale. Different SEM techniques, like EDS/WDS or TOF-SIMS would not be able to resolve the graphene. Thus, the Raman add-on it an important feature for such 2-dimensional materials which are in the focus of thousands of researchers at the moment.

Silicon thin film solar cell

Raman spectroscopy is able to visualize tiny differences in composition or modification changes of materials. A good example for such sensitivity is the analysis of a thin film silicon solar cell. The layers of such photovoltaic devices are made of silicon with different structure: the first layer is amorphous, which means that the atoms are not periodically ordered like in a crystal. The second layer undergoes a depth-dependent transformation from mostly amorphous to a mixture of nanoscopic amorphous and microcrystalline volumes. Each phase shows distinct Raman spectra and their ratio can be precisely determined. The depth-resolved phase composition is most important for the energy conversion efficiency of the solar cell and helps the engineers in improving their material. Furthermore, such a complex layered nanoscale material can be fully automatically analysed by subsequent FIB cutting and imaging to create a 3-dimensional model of the layers and its interfaces.

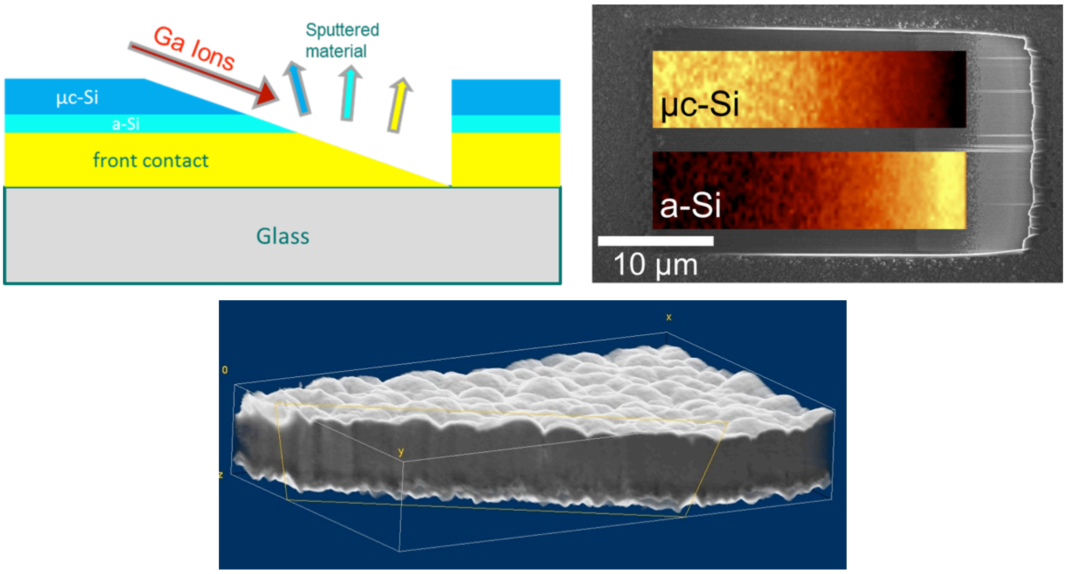

Figure 20: Upper left: Scheme of the ion beam preparation of a shallow wedge to enable Raman mappings with high depth resolution. Upper right: SEM image of such an etched wedge with Raman signal overlays for the microcrystalline and the amorphous silicon phase. Bottom: FIB-based 3D-reconstruction of a thin film solar cell to visualize the nanoscale roughness.

Industrial planar GaN LED

Comparable to the silicon solar cell, we frequently help industry partners in understanding their complex materials. The newest analytical tools enable the in-depth understanding in a yet unknown complexity.
An industrial producer of InGaN/GaN LEDs contacted us with a problem regarding a new prototype production machine. Photoluminescence measurements (figure 3 upper left) showed large differences over the wafer surface and they did not know what happened. We used the UnivSEM tool XEIA as ideal analysis instrument, since its Xenon plasma ion beam would not interfere with the Gallium of the layer, which would be a problem in a typical Gallium FIB. Different areas of the wafer were sputtered by the ion beam and the mass spectrometer analysed the sputtered material.

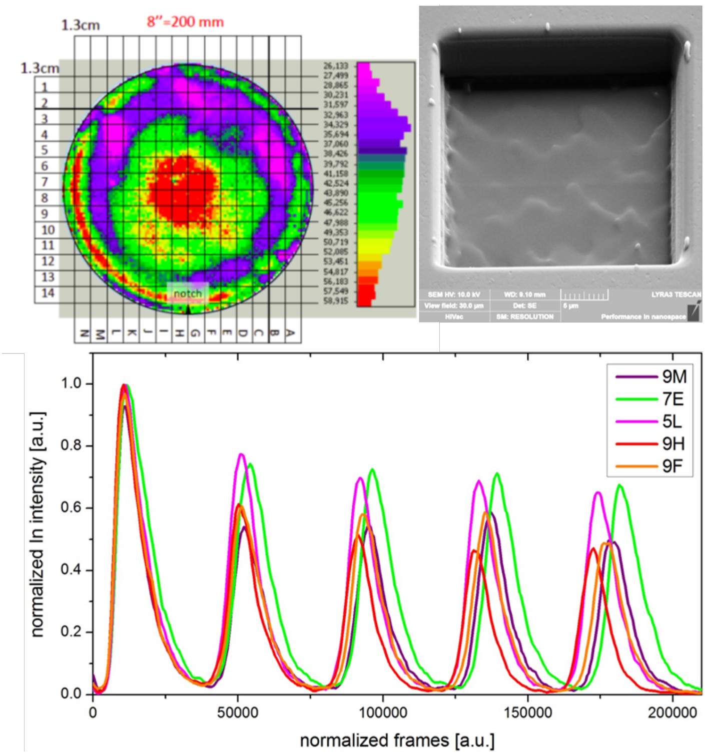

Figure 21: upper left: PL intensity map of an LED wafer from an industry prototype production. Upper right: TOF-SIMS crater etched into the layer stack. Bottom: TOF-SIMS measurements of Indium distribution at different positions of the wafer. The light-emitting quantum wells show differences both in intensity as well as position.

By focusing on the Indium content from five stacked InGaN so called 'quantum wells', which basically generate the light in this type of LED, we were able to show large differences in quantum well quality. After these measurements, the company realized that they had a problem with their temperature distribution, which lead to different growth velocities and thus hampered the LED quality.

Micro and nano-antennas

Micro – and nano-antennas were fabricated in the vicinity of nanostructures to be analysed using decomposition of metalorganic molecules (so called "precursors") by focused electron (so called Focused Electron Beam Induced Deposition - FEBID) and ion (Focused Ion Beam Induced Deposition - FIBID) beams, both produced in the UnivSEM tool. These antennas should provide enhancement of spectroscopic signals from the analysed nanostructures (e.g. nanowires, nanodots) localized on various substrates, and thus to increase the capability of spectroscopic methods to analyse such small objects as nanostructures. To show the capability of the UnivSEM microscope, TiO2 nanowires and ZnO nanoparticles were prepared on Si substrates outside of the microscope and then Pt nanoantennas were fabricated by the above mentioned methods Focused Electron and Ion Beam Deposition (FEBID and FIBID). This is demonstrated by the following figures:

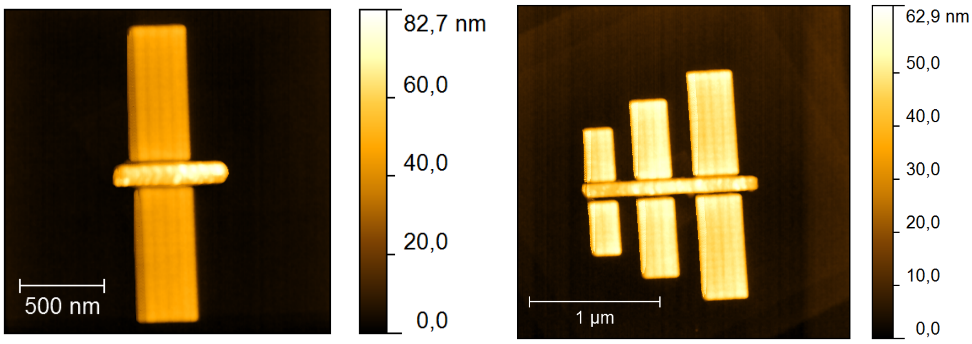

Figure 22: 3D topography image of TiO2 nanowires together with fabricated nanoantennas acquired by atomic force microscopy.

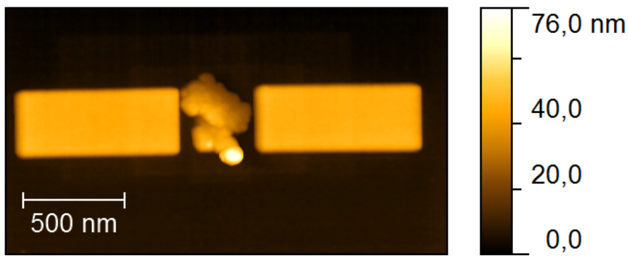

Figure 23: AFM topography image of the ZnO nanoparticle with a nanoantenna.

Comparative application studies

In this task the feasibility study of the potential incorporation of a special atomic force microscope (AFM) measuring optical signals in the vicinity of nanostructures (i.e. optical near fields) into UnivSEM has been done. As a part of this study, different experimental configurations of such a microscope, called scanning near field optical microscope (SNOM), have been proposed. In these configurations, the implementation of an optical fibre into a vacuum chamber of the UnivSEM for local illumination or signal collection is much simpler. The only component, which is necessary to install in this chamber is an AFM head for scanning of the optical fibre probe.

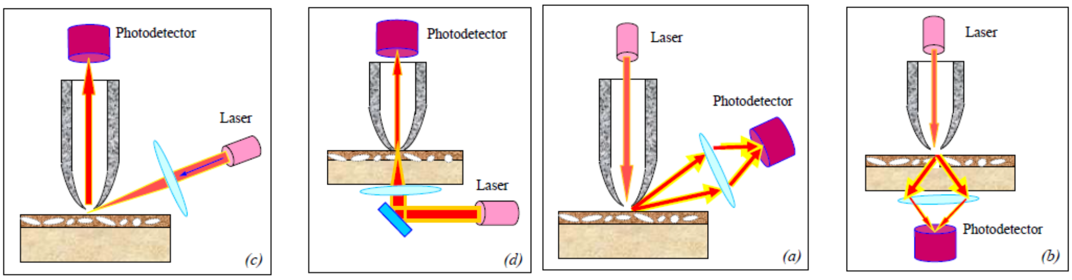

Figure 24: Possible configurations of a near-field optical microscope (SNOM). Two left pictures: illumination modes, two right pictures: collection modes.

Further, the ability of the SNOM for the study of optical near fields generated by metallic nanostructures has been verified experimentally. In Figure 25 the distribution of the optical near fields related to special electromagnetic waves confined to the Au surface (called surface plasmons) measured in the collective mode of SNOM is shown. To carry out the experiments, metallic structures consisting of slits milled into the Au film by Focused Ion Beam (FIB) were fabricated, a technique, which is available in the UnivSEM tool.

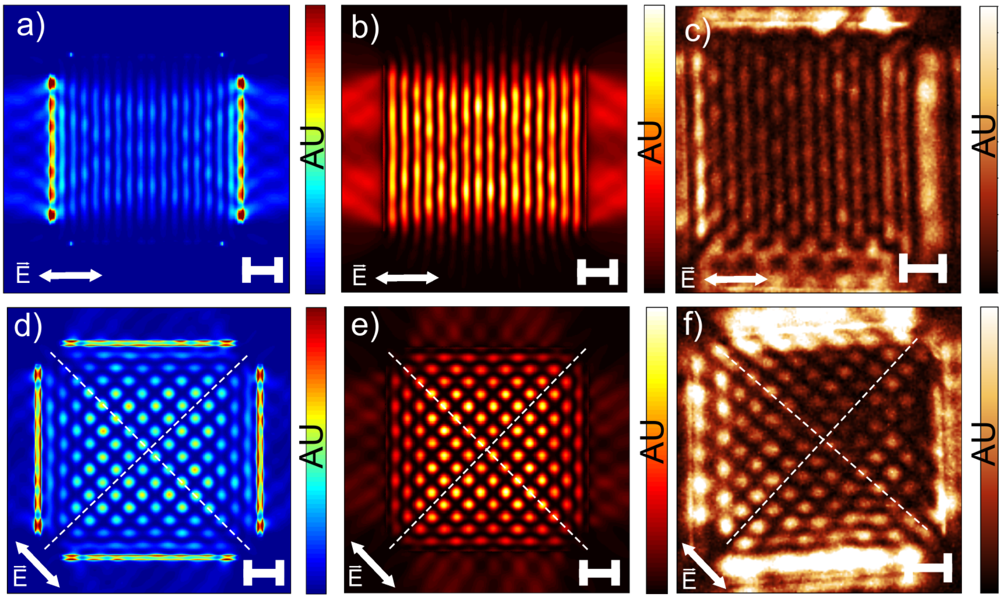

Figure 25: Numerical simulation (a), analytical calculation (b) and experimental SNOM image (c) of the optical near-field patterns established over the square-like structure being illuminated by the laser beam (λ = 630 nm).

We have measured the interference patterns at different wavelengths and modified the apparatus for spectroscopic measurements using a supercontinuum white light laser.
The achieved results demonstrate the ability of the method to spatially control the near-field energy distribution, which can be utilized in nanolithography, trapping and selective growth of nanoparticles, and other fields of nanotechnology and nanoscience.

4. The potential impact

Exploitation activities

UnivSEM is an industrially driven strategic project, which enables the coordinating company to co-develop a design of a unique tool and integrate the newly developed or improved components into a new system. It is expected that this new system (e.g. vacuum chamber) will become a new industrial standard for the years to come, which will enable users to adapt the analytical performance based on demand (or resources). This modular approach has been validated by the Advisory Board Members, early in the project. The design of the newly created vacuum chamber has been already exploited since early 2014 and company TESCAN ORSAY HOLDING, is wisely promoting it's deployment to their customers.

As part of the UnivSEM project, TESCAN ORSAY HOLDING, a multinational company experienced in charged particle optics and WITec, a distinguished German specialist in Raman and scanning probe microscopy, successfully introduced a correlative microscopy technique, which combines electron microscopy with Raman microscopy. The resulting RISE (Raman Imaging and Scanning Electron) Microscopy technique enables for the first time ultra-structural and chemical imaging with one microscope. The product was launched at Analytica – Trade fair for instrumental analysis, laboratory technology and biotechnology. About 18 press representatives joined the conference. The press was also interested in the presentation of EC project officer Dr. René Martins. Additionally to the press conference we distributed around 50 press kits at Analytica.

WITec was given a 2015 Photonics Prism Award for the correlative RISE microscope as winner in the metrology category. The Prism Award is given for top innovations in the field of photonics, granted by Photonics Media and sponsored by the international Society for Optics and Photonics (SPIE.). The winners were chosen from more than 130 applicants.

TOFWERK (SME): A version developed within the 6 first months of the project is already offered as a product. An updated version based on later UnivSEM developments is expected to be offered by end of 2015.

Dissemination activities
Project partners often present at the top scientific conferences and publish their results in scientific articles in high impacted journals. The project generated over 20 scientific articles and it was presented in over 30 scientific events and conferences.

Several dissemination materials were prepared at the beginning of the project (leaflet, roll-up). Roll up with essential information about the project was produced and was available for any project partner purposes.

The project objectives and recent results were presented during Industrial Technologies 2014 (9th – 11th April 2014, Athens). The project exhibition stand did not offer only information on the project itself, but also gave an opportunity to see the first demonstrations of its outputs (e.g. newly developed 4-axis stage, press releases and posters). More than 40 conference participants that showed their interest in the presented concept and prepared prototypes, visited the stand. The stand was also visited by delegation of members of the NMP Programme Committee. The introductory video was prepared for continuous loop presentation at the stand.

UnivSEM organized two dissemination events, one focused on advanced auditorium (potential users of UnivSEM tool) in Brno on 29th – 30th April 2014 and second focused on the informed public and policy makers in Brussels on 12th March 2015.

The Brno event was organized in the new premises of the Tescan Orsay Holding in Brno, Czech Republic. The participants were welcomed by the Chair of the Board of the holding, Mr. Jaroslav Klíma, who stressed the importance of the correlative microscopy for the future progress in the material research and in process development. The UnivSEM coordinator, Dr. Jaroslav Jiruše (TESCAN Brno, s.r.o) presented The New Multifunctional FIB-SEM Tool for Nanotechnology. His talk was followed by presentations of Olaf Hollricher (WITec GmbH), Oliver Schaff (SPECS Surface Nano Analysis GmbH), James Whitby (EMPA), Fredrik Oestlund (TOFWERK AG), Dietmar Vogel (Fraunhofer ENAS), Björn Hoffmann (Max Planck Institute for the Science of Light), Tomáš Šikola (Brno University of Technology), Alexander Korsunsky (University of Oxford), Patrick Philipp (CEA, Unit. d'Instrumentation Scientifique) and Helena Desciova (ON SEMICONDUCTOR Czech Republic, s.r.o.) The workshop participants had the opportunity to see several practical demonstrations and had possibility to discuss with the dedicated experts on all integrated technologies from UnivSEM project. The presentations from the workshop were made available on the UnivSEM webpage.

"The full UnivSEM tool was exhibited at International Microscopy Conference in Prague in September 2014. The interest of general microscopic public was high thus future prospects seem to be promising. The project coordinator Dr. Jaroslav Jiruše gave a lecture on project results in the scientific part of the conference."

The Brussels event was organized in CZELO – Czech Liason Office for Research, Development & Innovation. The event was designed as an afternoon event only and number of important policy makers from various regions and DG Research and Innovation participated to this event.

Based on the recordings performed during M24, 3 videos of UnivSEM project were prepared. The first, is a short teaser with main objectives described by the project coordinator. The second video is based on the explanation of partners roles and motivations in this project. The third video describes the technical outputs of the project. In the last 6 months (since the video has been made public – Univsem.eu and Youtube) it attracted more than 200 visitors.

MPL contributed to the featured interview "Highlights from 25 years of Nanotechnology":
http://iopscience.iop.org/0957-4484/page/25th-volume, Direct video link: http://bcove.me/11s3skbo

Last but not least the tool was also promoted in Organic Bioelectronics Linkedin group, as it could bring unique analytical capabilities to organic electronic in general and bio-interfacing electronics in particular.

5. The address of the project public website

http://www.univsem.eu

Main contacts:

Project coordinator
Jaroslav JIRUSE, PhD.
TESCAN Brno, s.r.o (Czech Republic)
jaroslav.jiruse(at)tescan.cz
+420 547 130 468

Project manager
Rudolf FRYCEK, PhD.
AMIRES Sàrl (Switzerland)
frycek(at)amires.eu
+41 796 784 295