Ion beam tools for nondestructive in-situ and in-operando composition analysis and modification of materials at the Tandem Laboratory in Uppsala

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ABSTRACT: Ion accelerators have demonstrated tremendous versatility in their research applications throughout several decades. Starting predominantly as nuclear physics tools, they subsequently provided novel insights into fundamental atomic physics. Nowadays small and medium size accelerators are routinely employed in several branches of materials science. As research questions increasingly drive the need for analyses and material modification experiments performed in-situ and/or in-operando, facilities must adapt to service such a need. In this work, we highlight capabilities for in-situ and in-operando composition depth profiling and materials modification, developed with instrumentation at the Tandem Laboratory at Uppsala University. An overview of available ion sources and accelerators is given. We then exemplify the specific capabilities of the available end stations for materials synthesis, sample modification and device operation either during simultaneous ion beam based characterization or with such characterization performed in-situ before and after the application of various modification steps. Describing specific recent examples and potential future applications, we illustrate the impact of the present national research infrastructure.

KEYWORDS: Accelerator Applications; Instrumentation and methods for time-of-flight (TOF) spectroscopy; Ion sources (positive ions, negative ions, electron cyclotron resonance (ECR), electron beam (EBIS))

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1 Introduction

Nondestructive ion beam analysis (IBA) has been available to support materials science with composition depth profiling of the near-surface regions in both thin film systems and bulk samples since the 1950s [1, 2]. When discussing the advantages of IBA, one typically compares it to other techniques that provide information on near-surface chemical composition, including X-ray fluorescence analysis (XRF), energy-dispersive X-ray spectroscopy (EDX), X-ray photoelectron spectroscopy (XPS), scanning Auger microscopy (SAM), secondary ion mass spectrometry (SIMS), laser induced breakdown spectroscopy (LIBS) and glow discharge optical emission spectroscopy (GDOES). With all these possibilities available, the unique advantage offered by IBA lies in the fact that it simultaneously, in addition to being comparatively nondestructive, provides depth-resolved and quantitative information, without the need for extensive sample preparation or specific calibration standards (see also the in-depth comparison of IBA with complementary techniques in [3]). As such it has found a wide range of applications, for example routine analysis of thin film material systems [3], measurements of trace element concentrations in the environment and in food [4, 5], characterization of historical artifacts [6] and investigations of the thickness and composition of deposited material layers in magnetic confinement fusion devices [7]. Analysis of materials that require specialized treatment, such as liquid and solid samples of volatile substances, has been demonstrated [8].

When it comes to thin film analysis, the miniaturization of various industrial products, in particular integrated circuits, has made systems of interest increasingly susceptible to alteration
during transport and storage. Components like thin film batteries alter their composition during operation, potentially making analysis in an unperturbed state insufficient for answering relevant research and design questions. Furthermore, the initial stages of decomposition of a material, e.g. oxidation, may well be of interest to study as they occur. In cases where the dynamics of a synthesis path are to be investigated, that synthesis must necessarily be performed in a set-up that also contains characterization instrumentation. These circumstances create a need for materials research carried out in a manner such that sample synthesis, modification and characterization are performed in a single setup (in-situ) or even while a device is being operated (in-operando). The characterization and modification tools need to be robust, operable for samples placed under specific working conditions and any analysis needs to have a minimal impact on the process under study. IBA methods are often suitable for fulfilling many or all of these criteria.

A recent topical collection of articles summarized a significant portion of the existing small and medium sized accelerator infrastructures in Europe [9]. When it comes to IBA facilities, laboratories at the Universities of Lisbon [10] and Florence [11], the Nuclear Physics institute of the Czech Academy of Sciences [12], the Institute of Nuclear and Particle Physics in Athens [13] and the Atomki Accelerator Centre in Hungary [14] among others were described. We complement those works here with a description of the Tandem Laboratory at Uppsala University, a Swedish national research infrastructure. We highlight the presently available instrumentation for in-situ and in-operando materials synthesis, modification and characterization through a detailed description of existing equipment, a discussion of research possibilities and an overview of recent applications.

2 Accelerators and supporting systems

The Tandem Laboratory research infrastructure includes accelerator systems used for material characterization and modification in three different energy intervals, which are described individually here. The largest of these, and most prominently applied for standard IBA measurements, is a 5 MV pelletron tandem accelerator model 15SDH-2, delivered by National Electrostatic Corporation (NEC), commissioned in 2001 and operated up to the rated maximum terminal voltage since shortly thereafter. During day-to-day operation, however, the terminal voltage is typically limited to less than 4.2 MV in order to avoid lengthy machine conditioning. A lower limit slightly below 1 MV is usually selected to minimize angular divergence of the beam inside the accelerator itself, and thus ensure an acceptably small number of ion impacts on the inner surface of the acceleration tube. By shorting out segments of the acceleration tube a lower terminal voltage can be selected while maintaining the critical electric field profile, allowing operation at decreased beam energy.

A layout of the laboratory is shown in figure 1. Negative ions are delivered to the tandem accelerator by four ion sources: two model 358 duoplasmatrons from High Voltage Engineering Europa (HVEE), for which the initial source of material is a gas, and two Cs sputter sources using solid targets, see for example [15]. One of the duoplasmatrons is equipped with a potassium charge exchange canal and reserved for production of $^3\text{He}^-$ and $^4\text{He}^-$. When it comes to the Cs sputter sources, one was constructed in-house and the other is the commercial multi-cathode source of negative ions by cesium sputtering (MC-SNICS), model 2JA045110, produced by NEC [16].

All ion sources connected to the 5 MV pelletron are operated with standard extraction voltages of 10–20 kV, and 40–60 kV preacceleration voltage, yielding 50–80 keV singly charged negative ions.
Figure 1. Layout of the laboratory housing the 5 MV pelletron tandem accelerator. For descriptions of the beamlines and experimental end stations depicted in the lower left corner of the image, see section 3.1.

at the input to the accelerator, although this value can be increased to above 100 keV if so desired. A typical current delivered to the accelerator for He isotopes is 150 nA, while the duoplasmatron without charge exchange canal provides $^1$H, $^2$H, and NH$_2$ for proton, deuteron, $^{14}$N or $^{15}$N beams usually at currents up to approximately 5 μA. $^6$Li, $^7$Li, $^{12}$C, $^{16}$O, $^{19}$F, $^{31}$P, $^{35}$Cl, $^{74}$Ge, $^{79}$Br, $^{107}$Ag$^-$, $^{127}$I$^-$ and $^{197}$Au$^-$ are examples of ions delivered by the sputter sources, in many cases at maximum currents of at least several μA, which are intentionally limited to an allowable value for minimizing damage to the acceleration tube. This maximum allowed current into the accelerator is selected based on the X-ray yield due to interaction between energetic ions and inner machine components with accompanying acceleration of secondary electrons, and ultimately limits the current received at a target.

The upper energy limit for ions delivered with significant beam current from the pelletron tandem accelerator is on the order of 50 MeV for heavy ions, and is determined by the maximal charge state that can be achieved when stripping the projectiles of electrons in a gas cell at the high voltage terminal. As an example, charge state 11+ is available in sufficient quantity to yield about 0.4 nA of 48 MeV projectiles to target when stripping $^{197}$Au with 4 MeV kinetic energy at the terminal, whereas charge states 12+ and a few steps above in that case yield decreasing fluxes that may be measured by counting individual particles. See also further information on charge distributions for heavy ions after stripping in gaseous and solid targets in e.g. [17]. Higher charge states, up to at least 25+ can be made available by an additional stripping stage after the accelerator [18], without further gain in beam energy. The lower energy limit for a given terminal voltage depends on the mass ratio of the desired projectile to the ion that is produced in the source. For example, at 1 MV terminal voltage, H$^+$ may be delivered to target at 1.07 MeV by starting from NH$_2$. 
Charge states and energies of accelerated ions are selected in a double focusing analyzing magnet from Danfysik, with a maximal field of $B_{\text{max}} = 1.5 \, \text{T}$, and the ions are steered to one of six beamlines with user end stations by a switching magnet with a maximal field of $1.4 \, \text{T}$. Given a bending radius of $r = 1270 \, \text{mm}$ in the analyzing magnet, the mass-energy product for ions directed towards the target is classically limited by

$$mE_k \leq q^2 \frac{B^2_{\text{max}} r^2}{2} \approx Q^2 \times 175 \, \text{MeV amu},$$

where $q$ is the actual charge of the projectile, and $Q$ is the charge state. Exceeding the rated field in the magnet, the value can be increased to approximately $Q^2 \times 185 \, \text{MeV amu}$ if necessary.

An ion implanter based on the model 1090 implanter with model 920 CHORDIS high current ion source (see e.g. [19, 20], similar to the model recently sold as 921A as of the writing of this overview) from Danfysik is the second system of interest. It has been operated at the Tandem Laboratory since its delivery in 2003. The 920 ion source can be operated in gas, oven or sputter modes yielding, in principle, the possibility to produce ions of any element that can be obtained as a gas, a solid target or a powder (in the latter case with an upper limit on evaporation temperature of approximately 900°C), either pure or as a compound. Positive ions are accelerated from the source by placing it at a standard extraction potential of 20kV relative to the rest of the high-voltage end of the set-up, which is in turn elevated to a maximal $310 \, \text{kV}$ post acceleration potential using a Cockroft-Walton multiplier. The extraction potential can be varied both above and below the standard value. Thus, normally, a maximal total accelerating voltage of $350 \, \text{kV}$ is provided (see also [21]). By either applying a deceleration voltage up to $15 \, \text{kV}$, or decreasing the extraction potential, a minimum energy to target of less than $5 \, \text{keV}$ is available. Molecular ions may also be used in order to decrease the kinetic energy per individual projectile atom. A brief discussion on the performance of this system is provided in [22]. In general, isotope selectivity is possible across the periodic table thanks to a relative mass selectivity of $\Delta m/m \approx 1/250$, as long as there are not overlapping mass numbers occurring in the beam from the ion source, and typical currents are on the order of several mA for fairly abundant isotopes. Three beamlines, out of which one is equipped with an electrostatic beam chopper to enable ion flight time measurements, are connected to the $350 \, \text{kV}$ implanter. These are described in section 3.2.

Finally, low energy ions ($E_k \leq 10 \, \text{keV}$) are delivered in a setup based on the Colutron G-2 ion gun [23], which in turn is a development of the design in [24]. The gun is operated with a gaseous source material, and produces currents of singly charged ions up to a nominal maximum of $20 \, \mu\text{A}$. Typical currents at the output of the source, however, are on the order of a few $\mu\text{A}$ depending on the ion species, and a mass selectivity of $\Delta m/m \approx 1/400$ is achieved. Acceptable beam properties can be maintained down to a minimum energy of approximately $500 \, \text{eV}$. The source is equipped with an Einzel lens for beam focusing and a Wien filter for ion velocity selection. These are followed by a beam chopper enabling flight-time measurements, an ion scattering chamber and a UHV sample preparation chamber; all described in further detail in section 3.3. The $350 \, \text{kV}$ ion implanter laboratory as well as the low energy set-up with scattering and preparation chambers are displayed in figure 2.

In addition to the systems introduced above, the Tandem Laboratory research infrastructure incorporates a compact accelerator mass spectrometer dedicated to radiocarbon dating [25], which is not included in this overview.
3 End stations compatible with in-situ & in-operando experiments

3.1 5 MV pelletron accelerator

3.1.1 SIGMA set-up

The Set-up for In-situ Growth, Material modification and Analysis by ion beams (SIGMA), with an original design presented in [26], is an ultra high vacuum (UHV) system in which the MeV ion beams provided by the 5 MV tandem accelerator may be applied to sample films grown in-situ by electron beam evaporation. Gases may be mixed and introduced in a controlled manner with simultaneous variation of the sample temperature from below room temperature to approximately 1000°C via active heating and cooling. Substrate cleaning, sputtering and ion implantation experiments are carried out using an ion gun operated up to a voltage of 5 kV, typically with $^1$H, $^2$H or Ar ions. Ion beam analysis data may be collected before, after as well as during all of these sample treatments. The latter is made possible in the case of sample annealing by the 2020 addition of a backscattering detector equipped with a layered foil with $5.0 \times 10^{17}$ at./cm$^2$ of C and $8.2 \times 10^{17}$ at./cm$^2$ of Au, significantly reducing the amount of light reaching the detector from the glowing sample surface.

Since the publication of [26], the set-up has been modernized with the addition of a second passivated ion implanted planar silicon (PIPS) diode detector [27] for light ion elastic recoil detection analysis (ERDA), yielding a total of 2 ERDA detectors and 1 for Rutherford backscattering spectrometry (RBS) inside the chamber. These are possible to relocate, without breaking vacuum, along a circular, horizontal trajectory around the sample, at distances of 15–17 cm depending on the goniometer position. The presence of 2 detectors for ERDA allows simultaneous measurements with different absorber foils at different recoil angles. In addition, the original residual gas analyzer (RGA) has been replaced with a Pfeiffer PrismaPro QMG 250 F1. An automated data acquisition and control system has been constructed for carrying out sequential experiment programs while generating time-resolved data logs of sample temperature, beam current, specific partial residual gas pressure at the gas analyzer position and total pressure in four positions as well as region-of-interest integrals from the spectra acquired with the IBA detectors [28]. The set-up in its current format is displayed in figure 3.

Figure 2. 350 kV ion implanter with beamlines and user end stations (left), and low energy ion scattering system (right). For details on the analysis equipment and experimental end stations (as well as the abbreviated labeling of these here), see sections 3.2 and 3.3. The labels “ACOL.” and “ANA” respectively indicate the low energy ion scattering and sample preparation chambers, further described in section 3.3.
Figure 3. SIGMA set-up with a selection of equipment highlighted. The load-lock chamber fits 4 samples on a rotatable holder. These are moved to the main chamber individually by means of a transfer rod. Two gate valves separate the main chamber from the load-lock, which is mounted on a movable trolley, enabling undocking of the latter without breaking vacuum on either side.

SIGMA has been used in several recent projects on thin film growth and modification [29], investigations of deuterium retention in tungsten [30] and a study of the behavior of low activation steel during annealing for applications in nuclear fusion research [31]. In a recent experiment, a bilayer film of $YH_2$ on top of $YHO$ on a C substrate was deposited in-situ and examined using RBS before and after in-situ $^{18}O$-oxidation. The obtained RBS spectra, reproduced from data presented in figure 2 of [32] are shown in figure 4. These exemplify how the SIGMA set-up enables studies of reaction kinematics and pathways by a combination of ion beam tools with reactive growth. The capability for obtaining concentration depth profiles of specific isotopes in individual process steps is crucial for such studies.

3.1.2 Multipurpose IBA chambers

The versatile IBA beamline at the Tandem Laboratory features three measurement chambers: one standard IBA chamber for high throughput time-of-flight (ToF) ERDA, particle induced X-ray emission (PIXE), elastic backscattering spectrometry (EBS), nuclear reaction analysis (NRA) and RBS measurements, one specialized chamber for ToF-ERDA, NRA, EBS, PIXE and RBS on large samples (for example wall tiles from fusion experiments, see e.g. [33–35]), and one chamber with space for creation of custom set-ups and demonstration experiments employed e.g. in undergraduate and graduate student education. The standard IBA chamber allows mounting of holders for up to 20 samples, which can be run automatically before manual switching of the holder is necessary. The samples can be tilted in two directions without moving the point of interaction with the primary ion beam. It is possible to mount several PIPS detectors in the chamber for gathering RBS data. In particular, up to such 4 detectors may be placed at $170^\circ$ backscattering angle, annularly distributed around the line defined by the incoming beam. Their recorded pulse height histograms can be
Figure 4. RBS spectra obtained before and after in-situ $^{18}$O-oxidation of YH$_2$-YHO bilayer on a C substrate in the SIGMA set-up. In addition to the appearance of an $^{18}$O peak, a reduction of the Y signal associated with the top layer, as well as an increase in the total Y signal width constitute measurable consequences of the oxidation. For experimental details, see [32].

processed individually or added to increase the effective detector solid angle coverage. Furthermore, an Amptek XR-100 Si drift detector (SDD) for PIXE, and a ToF-ERDA detector [36] are mounted at fixed positions. While the chamber provides excellent possibilities for automated IBA measurements, it offers limited capability for in-situ and in-operando experiments. It does, however, provide the possibility for ion channeling experiments with in-situ MeV ion irradiation or implantation.

Sample sizes up to approximately $20 \times 15$ cm$^2$, several cm thick are accommodated in the specialized chamber. Here, the point of interaction with primary ions can be scanned by moving the sample 20 cm along a vertical axis perpendicular to the beam, and approximately 3 cm along the remaining 2 axes. The sample is also freely rotatable around the vertical axis. Even larger samples may be mounted with some difficulty and limited possibilities for movement. A new ToF-ERDA detector using a gas ionization chamber for the energy signal was installed in 2016, in order to provide improved mass resolution for heavy recoils and lower susceptibility to radiation damage than a system employing a PIPS detector [37]. A stable pressure of approximately $4 \times 10^{-7}$ mbar is maintained in the detection system by active pumping when in standby, and it is typically in the low $10^{-6}$ mbar range during operation, depending on the vacuum quality in the scattering chamber itself. As a result of these relatively small pressure variations, detection efficiency variations can be kept acceptably small for extended operating periods (several months), which is not the case to the same extent in a system where the pressure during standby is uncontrolled.

A PIXE detector of the same type as in the standard IBA chamber is mounted at 90° angle from the forward beam direction in the specialized chamber. Feedthroughs for several input/output signals are also present, allowing ion beam analysis to be performed, for example, on electronic components in-operando. In a recent work, an array of PIPS detectors was set up along with a specifically designed sample holder, allowing coincidence IBA measurements in transmission and backscattering geometry to be carried out [38]. This set-up has been employed for in-operando
investigations of the Li and O distributions within a Li thin film battery stack while the battery was charged and discharged [39, 40]. Furthermore, in-situ and in-operando optical characterization and manipulation, exemplified in the case of photochromic \(\text{YH}_x\text{O}_y\) in [41], is enabled thanks to the presence of several large viewports.

### 3.1.3 Irradiation beamline

When operation of the present pelletron accelerator began in 2001, the MeV-ion irradiation beamline described in [42] was transferred to the facility. Since then it has been employed for various irradiation experiments investigating, for example, defects, vacancies and ion tracks in Si, \(\text{SiO}_2\) and SiC [43–45], and the effect of heavy ion irradiation on the photoluminescence of Si nanoparticles [46, 47]. During irradiation experiments the ion beam delivered by the tandem accelerator is scanned homogeneously over an area up to \(10 \times 10 \text{cm}^2\) on the target, and ion fluences up to a few times \(10^{16} \text{cm}^{-2}\) are readily achievable within hours of exposure time, depending on the selected projectile species. Samples are mounted on frames, which are installed in a magazine into a load-lock chamber and transferred to the irradiation position one by one. Wafers up to 4” in diameter are conveniently mounted on respective compatible frames. A recent refurbishment included a rebuilt sample transfer and position control system, modernized control software in LabView and improvement of the sample holder frames for increased system reliability [48]. A flange connection for mounting electrical feedthroughs is available behind the sample position, enabling for example in-operando studies of electrical properties while a bulk material or thin film sample is being irradiated.

### 3.1.4 Microbeam facility

At the scanning nuclear microprobe, a collimator followed by a focusing device of the Oxford coupled triplet configuration [49, 50] produces a beam spot with lateral dimensions down to slightly over \(1 \times 1 \mu\text{m}^2\) for p or He projectiles. The system is based on and rebuilt from the SLIM-UP microprobe described in [51]. A beam current high enough for NRA, PIXE and EBS to be performed (typically on the order of 100 pA) is maintained. The beam is scanned over a region of up to \(1 \times 1 \text{mm}^2\), enabling the creation of lateral concentration maps for elements of interest with depth selectivity. See also description in [52, 53]. The chamber contains a liquid nitrogen cooled lithium-drifted silicon (Si(Li)) X-ray detector and a 1500 \(\mu\text{m}\) thick annular charged particle detector at the center of which the beam enters the chamber. The shape of the scan area at the sample position may be observed using a fluorescent target and an optical microscope mounted at a fixed position. Recent applications include mapping of fuel atoms trapped in plasma-facing components from nuclear fusion devices [54] and determination of the sequence in which inks were deposited on paper [55]. Sample contacting via wire feedthroughs is possible, which makes in-operando mapping of e.g. current induced material migration possible. A potential application is in-operando studies of memristors, investigating the microscopic cause of memristic switching which is hypothesized to be connected to ion migration [56].

### 3.1.5 Nuclear reaction analysis set-up

This end station is equipped with a small target chamber kept at a pressure of approximately \(10^{-8} \text{mbar}\), and primarily employed for hydrogen depth profiling in material samples employing the resonant nuclear reaction \(^{15}\text{N}(\text{H},\alpha\gamma)^{12}\text{C}.\) The reaction features a sharp resonance in its cross
section at an energy of 6.385 ± 0.005 MeV for the $^{15}$N projectile and emits a characteristic gamma photon with an energy of 4.43 MeV [57, 58]. These gammas are recorded in a large solid angle, lead-shielded bismuth germanate (Bi$_4$Ge$_3$O$_{12}$) scintillation detector 10 cm from the target in the forward beam direction, covering a solid angle larger than 1 sr. An additional PIPS detector for RBS or EBS analysis is mounted at a fixed scattering angle of 160°. Automated sample motion, including rotation around one axis perpendicular to the beam, and remote control of the accelerator system allows stepping of the beam energy and ion channeling experiments to determine depth profiles and lattice positions of hydrogen atoms stored in solid materials [59]. A heat conductive structure in thermal contact with an outside reservoir allows cooling the sample to liquid nitrogen temperature or heating it above room temperature ($\leq 90^\circ$C). The addition of electrical feedthroughs is also possible, for in-situ voltage application.

A beamline for accelerator mass spectrometry constitutes the last end station at the 5 MV pelletron accelerator. It is not considered to be of interest in the present context, but is described in [60, 61].

3.2 350 kV ion implanter

3.2.1 Medium energy ion scattering chamber

The time-of-flight medium energy ion scattering (ToF-MEIS) beamline [22] has been in operation with the implanter as its source of energetic ions since 2012. It features a chopper with electrostatic deflection of the beam and sweeping in 2 dimensions across an exit slit to yield approximately 1 ns long pulses. These pulses can be further compressed in a drift tube buncher, down to approximately 0.3 ns and are then directed towards a sample to be analyzed. The sample is mounted on a goniometer compatible with translation of the sample along three axes as well as rotation around all of the same axes. To collect either scattered or transmitted particles, a large-area position sensitive microchannel plate (MCP) detector, located 290 mm from the sample and with an active area solid angle coverage of 0.12 sr is employed. It can be placed at any scattering angle along a circular track around the periphery of the chamber. The velocities of detected particles are measured by recording the time difference between a trigger pulse emitted by the beam chopper and the signal pulses received from the MCP. Thus, both standard MEIS measurements with a selected scattering angle interval, and ion channeling or blocking experiments that lead to characteristic patterns on the detector surface (see e.g. [62]) are possible to perform. The set-up further allows annealing [62–64] as well as implantation [65] in-situ.

As an example, figure 5 shows energy converted ToF-MEIS spectra and maps of the angular distribution of scattered ions on the large-area position sensitive detector, for a sample consisting of a 2.7 nm Ni film on top of a (100) oriented Si substrate. These were reproduced from data presented in [62]. A primary beam of 50 keV $^4$He$^+$ was employed with data collection for the energy spectra at a backscattering angle of 140°. Incident and exit angles were 15° and 25° with respect to the sample surface normal. The two spectra were acquired before and after in-situ annealing at 540°C. As a result of silicide formation, the Ni peak is broadened after annealing, and the Si signal edge is shifted towards a higher energy.

The angular distribution maps were obtained for scattered ion energies between 34 and 42 keV (corresponding to the signal from Ni), with an incident angle of 35° with respect to the sample
surface normal. This surface normal was pointing towards the center of the position sensitive detector at 145° scattering angle. The maps, acquired after annealing at 260°C and 540°C, demonstrate the difference between an amorphous or polycrystalline Ni film in the former case, and an epitaxial single crystalline, slightly off-stoichiometric NiSi$_2$ film in the latter. For the single crystalline film, a characteristic pattern appears due to blocking of scattered ions exiting the sample by the atom rows that constitute the crystal lattice.

![Figure 5](image)

**Figure 5.** Left — Energy converted ToF-MEIS spectra obtained with a 50 keV $^4$He$^+$ beam on a 2.7 nm Ni film deposited on a Si substrate, before and after in-situ annealing at 540°C. The modification of the spectrum is a result of silicide formation. Right — angular distribution of ions scattered from Ni, reaching the position sensitive detector. The image is split diagonally between the case of an amorphous or polycrystalline Ni film (homogenous distribution) after annealing at 260°C, and an epitaxial nickel silicide (showing a blocking pattern) after annealing at 540°C.

A second position-sensitive MCP flight time detector, with an increased flight distance of 1050 mm from the sample was installed at the ToF-MEIS chamber in 2019. It provides an improved energy resolution as compared to the large-area detector and is further described in [66]. It is centered at a scattering angle of 135°, covers a solid angle of 2 msr and features an electrostatic deflector for analysis of the scattered ions’ charge states (see also the scattering chamber layout in [66]).

### 3.2.2 Multipurpose nuclear reaction and scattering set-up

A separate beamline was recently connected to the ion implanter, featuring a measurement chamber for low energy PIXE and NRA as well as high resolution RBS [67]. The chamber was designed with the intention of utilizing two specific reactions: $^{11}$B(p,$\alpha$)$^8$Be and $^{18}$O(p,$\alpha$)$^{15}$N to depth profile B and $^{18}$O, the latter of which is especially relevant as a tracer isotope in material studies for nuclear fusion research [68]. In addition to a standard PIPS detector and an SDD PIXE detector, the chamber contains a cooled surface barrier detector with FWHM energy resolution of approximately 4 keV, based on a design by Geretschläger [69], at 135° backscattering angle. Like the ToF-MEIS setup, the multipurpose chamber is compatible with in-situ ion irradiation and/or implantation followed by IBA. Ion channeling experiments can also be carried out by tilting the sample around two axes perpendicular to the beam.
3.2.3 Ion implantation set-up

High-throughput ion implantation is performed in a set-up where the beam is scanned across the surface of a target. Wafers of 6 inch diameter or smaller are readily accommodated and mounted on a sample wheel with 6–8 positions depending on wafer size. Smaller samples can be clamped in groups to backing plates and exposed as needed. With a beam current to target of more than 100 μA, ion fluxes on the order of $10^{14}$ cm$^{-2}$s$^{-1}$ are attainable. The setup enables the target to be mounted on a contact heater for in-situ annealing to $\leq 650^\circ$C. During high-current implantations, cooling may be applied using pressurized air.

3.3 Low energy ion scattering system

In 2018 the *set-up for Analysis of the Charge Of Light Ions Scattered from Surface Atoms* (ACOL-ISSA [70]) was transferred to the Tandem Laboratory infrastructure from the Johannes Kepler University, Linz, Austria. The set-up includes a sample preparation and analysis (ANA) chamber originally described in [71]. A base pressure of approximately $4 \times 10^{-10}$ mbar is maintained in the ANA chamber by means of a single turbo pump as well as a Ti sublimation pump and a liquid nitrogen cold trap. A three-cell evaporator, Omicron model EFM 3T operable with material sources in the form of rods or powder-loaded single crystal Mo crucibles, allows in-situ sample growth. The diameter of the spot on the target sample within which evaporated material is deposited can be varied between approximately 8 and 15 mm. Up to three materials may be evaporated simultaneously, and an integrated flux monitor enables calibration of the deposition rate. The possibility for ion sputtering at energies up to 5 keV and at a typical current of a few hundred nA is provided by a Physical Electronics Industries (PHI) model 04-191 sputter gun. While the beam produced by this unit has a typical spot size of approximately 3 mm across, it can be rastered to uniformly erode an area up to on the order of a square centimeter. In addition, the sample can be annealed at temperatures up to approximately 1000°C, and gas exposure at a controlled pressure may be performed.

Analysis options in the ANA chamber are granted via the presence of a PHI model 10-155 Auger electron spectroscopy (AES) set-up and an ErLEED 3000 unit from SPECS GmbH for low-energy electron diffraction (LEED). Electron acceleration voltages (typical beam currents) are 0–5 keV (30 nA) for the AES set-up and 0–1 keV (up to 2.5 μA) in standard LEED operation. Further analysis with time-of-flight low energy ion scattering (ToF-LEIS) is performed in the ACOLISSA chamber, to which samples are transferred without breaking vacuum by means of a UHV-compatible manipulator. An electrostatic chopper (see details in [70]) is here used to produce beam pulses with lengths down to approximately 20 ns, and ions scattered from the sample are recorded with an MCP detector centered at a scattering angle of 129°. The detector is circular with an active area of 18 mm diameter, at a flight distance of approximately 650 mm from the sample. Similarly as described for the ToF-MEIS setup above, the scattered ion velocity is determined based on the time difference between the chopper pulse and the arrival time at the MCP detector. Furthermore, application of a post acceleration voltage separates the signals pertaining to ions and neutrals scattered from the target. While the ion signal provides information about the sample surface (down to a single monolayer depending on the experimental conditions [72]), compositional data in a depth interval of several nm can be extracted from the neutral signal. The ACOLISSA chamber is also compatible with in-situ annealing up to 800°C in a base pressure of $2 \times 10^{-10}$ mbar.
The capability for in-situ sample growth and characterization provided in the ANA-ACOLISSA system has previously been utilized for investigations of ultrathin layer growth \cite{73} and electronic energy loss of keV H and He ions in thin films of Al, Ni, Cu, Ta and Au, grown both ex-situ and in-situ \cite{74–76}. Recent work includes a study of the dynamics of Nickel silicide formation for contact layers in solid state electronics \cite{77}, and thermal segregation of alloying elements to the surface of reduced activation nuclear steel \cite{78}, both relying on in-situ annealing.

Figure 6, reproduced from data presented in \cite{73} shows examples of energy converted ToF-LEIS spectra obtained with 1.2 keV $^4$He$^+$ probing ions on thin Au films grown in-situ on top of in-situ B-coated Si substrates. Post acceleration was applied to separate the signals from ions and neutrals. The nominal film thicknesses were 1 Å and 32 Å, and TRBS \cite{79} simulations based on these thicknesses are overlain for comparison with the neutral signals. The dashed vertical line indicates the surface single scattering energy, while the overlain depth scale gives the depth of origin of an ion with the corresponding exit energy assuming straight line incoming and outgoing trajectories, single scattering and energy loss evaluated at the initial energy based on the data presented in \cite{75}. Note that this procedure only provides an approximate depth scale, as the assumptions of single scattering and straight trajectories become inaccurate even just a few monolayers below the sample surface. As the ion signal for the nominally 1 Å thick Au film is significantly less pronounced than that of the 32 Å film, it is deduced that the former is in fact not completely covering the surface, but rather consists of separate islands. The signal obtained from backscattered neutrals contains additional information about the Au present below the surface for the 32 Å film. The comparison with the TRBS simulation reveals an excellent agreement in the 32 Å case, indicating a high-quality thin film. In contrast, to fit the experimental spectrum for 1 Å nominal deposited thickness, a simulation for an 8 Å film provides the correct width, while the intensity has to be reduced by a factor of 0.18, again indicating island growth of the deposited Au.

**Figure 6.** Energy converted ToF-LEIS spectra with post acceleration, obtained on Au films grown in-situ on a B coated Si substrate. TRBS simulations for the nominal film thicknesses of 1 Å and 32 Å are overlain.

*The TRBS simulation for 1 Å nominal thickness was performed for an 8 Å film, and the resulting yield for each channel was multiplied by 0.18 to reproduce the measured spectrum. The procedure is compatible with the expected treatment required to reproduce the spectrum obtained on a sample with incomplete surface coverage, i.e. island growth of the deposited layer.
AES spectra obtained at different nominal thickness steps during the growth of the 32 Å Au film are shown in figure 7 to exemplify the possibility for in-situ characterization in the ANA chamber. Vertical solid and dashed lines respectively indicate the most prominent Auger electron energies for B and Au [80]. A continuous reduction of the B signal and increase of the Au signal are observed as the film grows. Altogether, these results demonstrate the potential of the system for in-situ characterization of samples otherwise highly susceptible to alteration by exposure to ambient atmosphere prior to characterization.

![AES spectra](image)

**Figure 7.** Auger electron spectroscopy performed on an Au film grown on a B-coated Si substrate at a range of nominal thickness steps. Prominent Auger electron energies are indicated by solid (B) and dashed (Au) lines.

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