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IBC - ION BEAM CENTER

Helmholtz-Zentrum Dresden – Rossendorf (HZDR) *

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Abstract: In the Ion Beam Center (IBC), various set-ups – electrostatic accelerators, ion implanters, plasma-based ion implantation equipment, low-energy ion tools, an ion microscope etc. – are combined into a unique facility for research and applications using ion beams. Almost all ions from stable chemical nuclides are available in the ion energy range from 10 eV to about 60 MeV. In addition to broad beams, also focused (down to 1 nm) and highly-charged (charge state up to 45^{+}) ion beams, or ions extracted from a plasma can be provided. In total, the IBC operates more than 30 dedicated tools or beamline end-stations. The specific expertise of IBC is the modification and analysis of solids by energetic ions aimed to develop novel materials for information technology, electronics or energy systems. In addition, ion beam analysis techniques became of increasing importance for interdisciplinary fields like geochemistry, climate or environmental research and resources technology. Special add-on services offered ensure a successful realization of user experiments. Based on a long-term expertise, specific equipment and common commercial procedures, the IBC is strongly active in the use of ion beam techniques for industrial applications aimed to initiate valuable product innovation.

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1 Overview

The Ion Beam Center (IBC) at the Helmholtz-Zentrum Dresden - Rossendorf (HZDR) is an international large-scale user facility for research and applications using ions beams which refers to many years of scientific and technical expertise in this field. In the IBC, various set-ups – electrostatic accelerators, ion implanters, plasma-based ion implantation equipment, low-energy ion tools, an ion microscope etc. – are combined into a unique facility. The available ion energy range varies from 10 eV to almost 60 MeV which corresponds to an interaction depth in solids between typically 0.1 nm and 10 μm, or some hundreds of micrometers for light ions. In addition to broad beams also focused (down to 1 nm) and highly-charged (charge state up to 45^{+}) ion beams, or ions extracted from a plasma can be provided. In total, more than 30 dedicated tools or beamline end-stations are available whereas up to ten experiments can simultaneously be performed. A schematic overview of the IBC is given in Figure 1.

Being a part of the Institute of Ion Beam Physics and Materials Research at HZDR, the specific expertise of IBC is the modification and analysis of solids by ions aimed to develop novel materials for information technology, electronics or energy systems. However, ion beam analysis (IBA) techniques are also of increasing importance for other fields like geochemistry, climate or environmental research and resources technology. As a "universal tool" for surface modification, ion beams hold a great potential for industrial applications which is actively supported at the IBC by ion beam services.

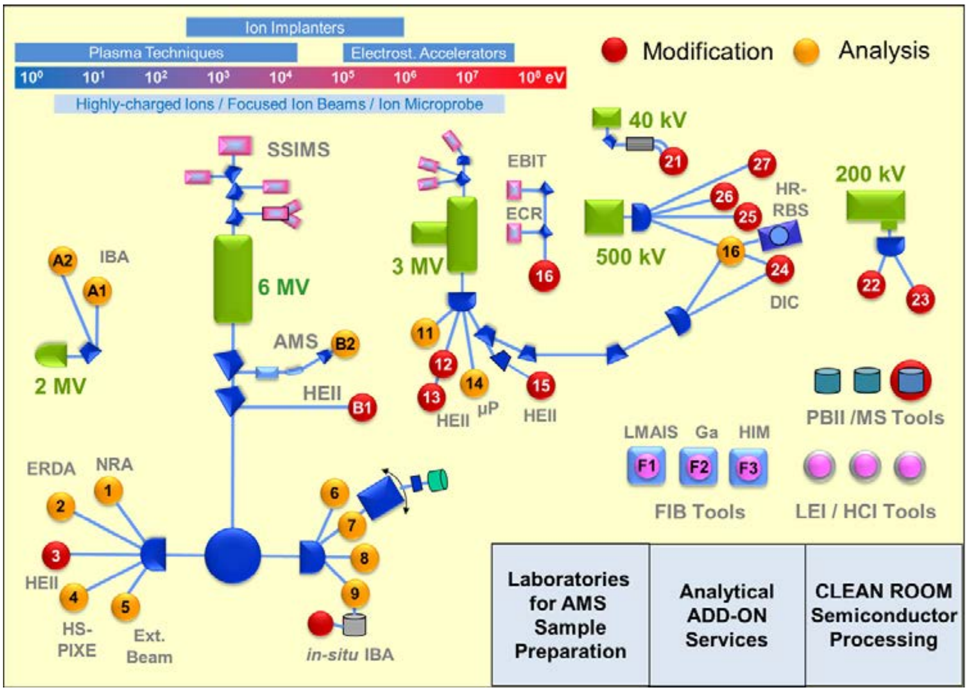

Figure 1: Layout of the Ion Beam Center, the color code illustrates various classes of tools / end-stations with respect to different user experiments. Further information and end-station descriptions are given at the IBC homepage http://www.hzdr.de/IBC.

Users of the IBC can take advantage of sophisticated add-on services to ensure a successful realization of their experiments. These add-on services include sample preparation and processing under clean-room conditions, structural analysis by e.g. electron microscopy / spectroscopy or X-ray investigations, specific software tools for the simulation of ion processes, or the support during data analysis and evaluation. These services are limited to an appropriate usage of resources and are offered free-of-charge only in combination with an allocated IBC experiment. Detailed information about the IBC, experimental equipment, and access rules can be found on the IBC homepage: http://www.hzdr.de/ibc.

In the following sections, different groups of ion beam techniques and equipment available at the IBC are described more in detail with respect to their capabilities and specifications.

2 Ion beam modification by ion implantation / irradiation

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The IBC operates three high-energy electrostatic accelerators (6 MV tandem, 3 MV tandem, 2 MV Van-de-Graaff), three ion implanters (with 40, 200 and 500 kV terminal voltages, respectively) and some stand-alone machines for plasma-based ion implantation. At accelerators or ion implanters, almost the entire periodic table of elements – but limited to stable isotopes – can be implanted very precisely in any solid target material with tunable depth and concentration profiles. At one specific end-station ion beams from the 500 kV ion implanter and the 3 MV tandem accelerator can be combined which enables double-beam irradiation or simultaneous irradiation / in-situ analysis studies. Basic parameters for ion implantation / irradiation experiments are given in Table 1.

Table 1: Basic parameters of ion implantation / irradiation facilities at IBC

Parameter	Value	Remarks
ion species	H to Au @ tandems H to Bi @ implanters	polyatomic implants possible at ion implanters, no implantation of radionuclides
ion energy	100 eV – 60 MeV	implanters: 100 eV – 1 MeV accelerators: 200 keV – 60 MeV
beam current	pA – mA	depends on ion species, ion source, specific end-station and experimental requirements
ion fluence	10^7 – 10^{18} /cm^2	depends on ion species and machine
sample size	mm^2 - ∅ 150 mm ∅ 200 mm @ 6 MV	from small samples to standard wafers
incident angle	0° / 7° fixed, or variable	variable tilt angles (0° - 85°) available at some end-stations; twist angle usually not specified
cooling, heating	water, liquid nitrogen, up to 1100°C	500°C (max. 3” wafer), 1100°C (1” sample)
vacuum	< 2x10^{-6} mbar	implantation / irradiation under ultra-high vacuum conditions (< 1x10^{-7} mbar) only on request
special features	fluence gradients, sample rotation	available only at certain end-stations

Ion energy and fluence values can be ensured with an uncertainty < 5%. A specific advantage of ion implantation / irradiation is that materials states far from thermodynamic equilibrium can be realized. These non-equilibrium states can be stabilized under certain conditions, or – if desired – relaxation into thermodynamic stable, steady-state systems is achieved as a result of subsequent sample processing (e.g. heat treatment).

Accelerators and ion implanters are mainly used for ion beam modification or ion beam analysis (see Section 3) of solid materials. In the field of ion beam modification typical applications are chemical doping and defect generation (Prucnal et al., 2015; Zhou et al., 2016), ion beam synthesis of nano-particles embedded in a matrix, surface functionalization (e.g. for biocompatibility), self-organization of surface nanostructures for templates, ion-induced disordering for phase transitions, materials and device damage generation for radiation tests, track/pore formation and many others. Plasma-based ion implantation using gas ions extracted from a low-pressure plasma offers specific advantages with respect to high-fluence implants (typical ion flux: 10^{15} ions/cm^{2}s) or surface modification of arbitrarily shaped (3D) objects.

Moreover, electrostatic accelerators are also applied for cross-section measurements of astrophysics reactions (Schmidt et al., 2014) or experiments related to the development of novel dosimeter or beam-diagnostic materials and radiation sensors.

3 Ion Beam Analysis

IBA responsible:

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Ion beam analysis (IBA) techniques are based on the interaction of high-energetic ions with sample atoms and the measurement of mass and energy of scattered, recoiled / sputtered ions and /or emitted secondary radiation. At IBC's electrostatic accelerators, almost all standard IBA techniques for non-destructive and quantitative (often standard-free) chemical analysis of samples with additional element-specific depth profiling up to a depth of several micrometer are available (see Table 2).

Table 2: Basic parameters of IBA techniques available at IBC

Method	Elements	Information depth	Detection limit	Depth resolution	Lateral resolution
Rutherford back-scattering spectrometry (RBS)	O - U	1 μm	0.1 at%	15 nm	0.5 mm
High-resolution RBS (HR-RBS)	Al - U	0.5 μm	1 at%	2 nm	1.0 mm
Elastic recoil detection analysis (ERDA)	H, B - Si	0.5 μm	0.1 at%	15 nm	1.5 mm
Particle induced X-ray emission (PIXE) • Standard set-up • PIXE camera • Ion microprobe	Si - U	some μm	0.001 at%	no distinct depth resolution	 mm < 100 μm 3 μm
Particle induced γ-ray emission (PIGE) • Standard set-up • Ion microprobe	low Z nuclei (Z < 15)	some μm	0.02 at%	no distinct depth resolution	 1.0 mm 3 μm
Nuclear reaction analysis (NRA)	H, B, C, N, O, F, ...	2 μm	0.02 at%	5 nm	0.5 mm

In addition, the IBC offers high-depth-resolution Rutherford backscattering spectrometry (HR-RBS), laterally-resolved elemental analysis, either by an ion microprobe or by a PIXE camera and in-situ analysis capabilities during sample processing. At some end-stations several IBA techniques can be simultaneously applied (so called "total IBA"), enabling an extended characterization of the sample according to the users' requests. IBA techniques are preferably used if the specific strength of these methods - the non-destructive, quantitative capabilities and the high depth resolution - are of particular importance. Typical applications are quantitative depth profiling of layered materials (Werner et al., 2016; Wilsenach et al., 2017), hydrogen content or depth profile measurements (Bilek et al., 2015; Jang et al., 2017), elemental mapping of geological or biological materials (Hanf et al., 2016) as well as fluence calibration for ion implantation doping.

4 Accelerator Mass Spectrometry

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For the determination of long-lived radionuclides decay counting is time-consuming or sometimes even impossible. Accelerator mass spectrometry (AMS) is superior since it does not rely on the disintegration of the radioactive nuclei. Using a high-energy accelerator it allows for the reduction of background and interfering signals, resulting from molecular ions and ions with similar masses, e.g. isobars. Thus, AMS generally provides much lower detection limits in comparison to conventional mass spectrometry. Lower detection limits extend applications to shorter and longer time-scales and to sample types that could never be investigated before. Nevertheless, basic, but accurate radiochemical sample separation is an essential prerequisite for AMS measurements. The DREAMS (DREsden AMS) facility, which is part of the IBC using the 6 MV tandem accelerator, offers excellent chemical sample preparation and measurement capabilities (Akhmadaliev et al., 2013). An overview of nuclides investigated at DREAMS and key information is given in Table 3.

Table 3: Radionuclides measured at the DREAMS facility

Radio-nuclide	Half-life of radio-nuclide	Radionuclide/ stable nuclide ratio	Measured ratios of nuclides	Machine blank level
^{7}Be	53 d	^{7}Be/^{9}Be	(0.01 - 2) x 10^{-12}	3 x 10^{-16}
^{10}Be	1.387 Ma	^{10}Be/^{9}Be	(0.01 − 300) x 10^{-12}	5 x 10^{-16}
^{26}Al	0.705 Ma	^{26}Al/^{27}Al	(0.001 − 60) x 10^{-12}	5 x 10^{-16}
^{36}Cl	0.301 Ma	^{36}Cl/^{35}Cl	(0.007 − 700) x 10^{-12}	4 x 10^{-16}
^{41}Ca	0.104 Ma	^{41}Ca/^{40}Ca	(0.006 − 9000) x 10^{-12}	20 x 10^{-16}
^{129}I	15.7 Ma	^{129}I/^{127}I	(0.5 − 200) x 10^{-12}	200 x 10^{-16}
Actinides	various		under development	

AMS is of utmost importance for dating samples on geological time scales. The spectrum of samples ranges from ice cores (Zipf et al., 2016), meteorites (Ott et al., 2014), deep sea manganese nodules and sediments, boulders from rock falls, landslides (Schwanghart et al., 2016) and glacier moraines (Landgraf et al., 2016) to lava samples from volcanic eruptions. DREAMS is also essential to research fields like astrophysics, climate, cosmo-chemistry or hydrogeology (Müller et al., 2016). Moreover, it is of significant interest and often used for nuclear decommissioning and safety, forensics and safety, dosimetry, nutrition, pharmacology, radioecology and toxicology research. The advantages of AMS, compared to other ultra-sensitive analysis methods, are an easier and faster sample preparation, smaller sample sizes, higher sample throughput and, therefore, largely reduced costs.

5 Facilities for low-energy and highly charged ions

Facility scientist:
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The IBC operates four stand-alone devices delivering ion beams of low-energy and / or highly-charged ions. For these kinds of experiments mostly (but not exclusively) ions from noble gases are used. For highly-charged ions, both electron beam ion traps (EBIT) (for highest charge states up to 45^{+}) and an electron cyclotron resonance (ECR) ion source (for medium charge states, typically $5^{+} - 14^{+}$) are in operation. An overview of the different facilities is given in Table 4.

Table 4: Basic parameters of LEI / HCI tools available at IBC

Equipment	Acronym	Ions	Energy range	Charge state	Ion flux
Low-energy ions	LEI	H – Xe	20 eV – 20 keV	typ. 1^{+}	$(10^{13} - 10^{15})$ ions/(cm^{2}s)
Highly-charged ions	HEI	He - Xe	20 eV – 20 keV per charge	3^{+} - 45^{+}	$(10^{9} - 10^{12})$ ions/(cm^{2}s)

Low-energy ions (LEI) with energies in the range of 100 eV to 25 keV interact only with the top few nanometers of a solid without affecting the bulk properties. Thus irradiation with LEI can be used to change the morphology and the properties of surfaces and thin films. In this energy range sputtering (i.e. the emission of atoms from the surface) is very effective and leads to the formation of different surface nano-patterns depending on the irradiation conditions, e.g. ion mass, energy, flux, fluence, incidence angle, and sample temperature (Keller & Facsko, 2010). For selected parameters extremely regular, periodic pattern are produced with periodicities in the range of 20 nm – 200 nm. These patterns can be either isotropic, i.e. hexagonally ordered dot or hole patterns (Keller & Facsko, 2010; Ou et al., 2015) or anisotropic as ripple patterns on substrates, which can be used as templates to grow nanostructured thin films with tunable properties.

Slow, highly-charged ions (HCI), on the other hand, are special in that they carry additionally a high amount of potential energy – the stored ionization energy – which is released and deposited into the electronic system of the materials surface (Gruber et al., 2016; Wilhelm et al., 2014). The electronic excitation by the potential energy of HCIs induces nanostructures on surfaces, in thin-films, or 2D materials by single ion impacts. Depending on the materials properties single hillock or pit structures are observed for every ion impact (El-Said et al., 2016). Common to the formation of these structures is a threshold in the potential energy due to the fact that the locally induced electronic excitation has to be high enough in order to induce a phase transition, like local melting or sublimation of the material.

6 Focused Ion Beam Techniques

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Focused ion beam (FIB) tools became the work horses of many R&D projects in academia as well as industry. However, standard Ga based liquid metal FIB systems are often used for TEM sample preparation only. This contrasts by the fact that many more ions can be provided in FIB and allow for research projects that go beyond the typical TEM lamella preparation. While a large number of metal and semiconductor ions can be extracted from liquid metal alloy sources recently also high-resolution noble gas based FIB systems became available. The latter help to avoid metal contamination of the samples and pushed the resolution limit for nano-fabrication into the single digit Ångström regime.

At the IBC three focused ion beam (FIB) systems are operating for ion microscopy and analysis, local surface or thin-film modification, nano-patterning or doping, as well as for sample preparation:

- Cross-beamTM workstation NVision 40 equipped with Gemini SEM and FIB column (Ga),
- Ion microscope ORION NanoFab working alternatively with He or Ne ions,
- Mass separated FIB with Canion 31M+ column using liquid metal alloy ion sources.

Basic parameters of the FIB systems at IBC are summarized in Table 5.

Table 5: Basic parameters of FIB systems available at IBC

Parameter	**Cross-beam FIB/SEM**	**Ion microscope**	**Mass-separated FIB**
ions	Ga	He, Ne	Si, Ge, Ga, Co, Ni, Nd, Er, Au, Bi; poly-atomic ions: Au_n, Bi_n + other ions on request
ion source	liquid metal ion source (LMIS)	gas field ion source (GFIS)	liquid metal alloy ion source (LMAIS)
energy	5 – 30 keV	5 – 35 keV	10 – 60 keV
spot size (resolution)	7 nm (at 1 pA)	He: 0.45 nm Ne: 1.8 nm	10 – 100 nm
ion current	1 pA – 45 nA	0.1 – 200 pA	1 pA – 10 nA
sample size	typ. 1 cm^2, 4” max.	~1 cm^2, typically	various, max. 6” wafers
additional features	• gas injection system • EDX, EBSD • pattern generator • TEM lamella preparation and lift-out	• gas injection system • electron flood gun • sample heater • in-situ 4-probe for electrical measurements • TOF based micro- RBS and SIMS	• sample heating and tilting • 6” laser interferometer stage for nm-scale positioning and stitching • electron flood gun

Besides application to fundamental and applied studies of materials science problems the focus is on the development, applications and equipment that go beyond state-of-the-art Ga-FIB techniques. The in-house research is focused on the development of new analytical methods, instrumentation for in-situ experiments and the investigation of new ion sources (Bischoff et al., 2016). Furthermore, these methods and procedures are used to investigate a wide range of ion material interactions in the fundamental as well as applied part of materials research (Hlawacek et al., 2014; Philipp & Bischoff, 2012). Recent projects include the fabrication of arbitrary sized nano-magnets using low-fluence focused Ne^+ ion irradiation. The minimal size of approximately 20 nm for these magnets is only limited by the size of the ion collision cascade. Broadening of the created geometries due to the beam can be excluded as the beam diameter is only 2 nm. In a different project semiconductor surfaces are patterned locally with the help of heavy poly-atomic focused ion beams. Recently, we also developed the first time-of-flight backscatter spectrometer for the gas field ion source (GFIS) based Orion NanoFab (Klingner et al., 2016). With an energy resolution of 2.5% and an unprecedented lateral resolution of 50 nm it can be used for spatially resolved, damage free materials analysis at the ion microscope.

7 Add-On Services

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For electron microscopy
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For AMS sample preparation:
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Many users make advantage from ion implantation / irradiation effects for the modification of properties of materials surfaces or near-surface regions. This is usually one step in a sequence of investigations ranging from materials synthesis to proof-of-principle tests for the envisaged applications. The users are very often interested to get fast information concerning the change of structural / physical / chemical properties of their samples after ion beam processing. For that reason, in combination with an allocated experiment the IBC offers a couple of ad-on services as listed in Table 6:

Table 6: Add-on services offered by IBC

Add-on service	Processes / techniques and major equipment / codes
samples preparation and processing	• sample cutting, polishing, cleaning, etching • annealing (ns – hours times regime) • lithography (optical and E-beam) • device processing
measurement and characterization	• optical and electrical characterization • electron microscopy / spectroscopy (SEM, TEM, AES, XPS): Hitachi S-4800 (SEM), NVision 40 (SEM/FIB), FEI Titan 80-300 (TEM), FEI Talos F200A (TEM), Microlab 310F (AES/XPS) • X-ray investigations Bruker D8 (XRD/XRR): Siemens D5005, Panalytical Empyrean, GE XRD 3003HR
AMS samples preparation	• two specific AMS chemistry laboratories for different nuclei available; chemistry preferably for nuclei of Table 3
data analysis and simulation tools	• simulation of ion-solid interaction (doping-, defect-, mixing profiles) codes: TRIDYN, TRI3DYN, kinetic Monte Carlo (phase separation) • simulation of surface patterning (home-made codes) • data evaluation of IBA spectra

The add-on services at IBC include analysis by complementary techniques, samples preparation or even device processing. For AMS investigations, the chemical preparation of AMS samples is mandatory and of key importance for reliable measurements. Moreover, in particular for IBA measurement, IBC users from interdisciplinary research fields have often less or no experiences with these specific techniques. Thus, the support during experiment preparation, measurement, data analysis and interpretation is essential to ensure an adequate result from the experiment and to satisfy widely users' expectations.

8 Specific equipment and procedures for ion beam services

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Ion beams as a "universal tool" for materials modification also hold a great potential for industrial applications due to their outstanding capabilities to change chemical, electrical, optical or other materials properties. To further extend the economic value of ion beam processing in industry, IBC facilities at HZDR are available for industry (research or ion-beam services) in close collaboration with the spin-off company HZDR Innovation GmbH and partners of the Geokompetenz-Zentrum Freiberg e.V. Such activities are based on individual contracts (usually quotation /offer procedures) with the partners that include the performance, delivery time and costs. In cases of well-defined and routine use of IBC facilities, long-term frame contracts are possible which makes the realization of the requested service easier, faster, more reliable and cost effective. With respect to decades of expertise in ion beam services, the IBC is well-familiar with common procedures required for collaborations with industry. This includes product qualification, ion beam processing in product lines, quality control, certification issues or the availability of specific equipment for large-volume processing. Actually, ion beam services cover mainly the following fields:

- High-energy ion irradiation of circuits for power electronics to improve the switching properties based on minority carrier lifetime engineering,
- Standard and high-energy ion implantation of doping elements for optoelectronic devices, power semiconductors, lasers or detectors to tailor the electric field distribution in such devices,
- Surface engineering by low- energy and plasma-ion-implantation to tailor / optimize specific materials' properties (e.g. high-temperature oxidation resistance, surface hardness, bio-compatibility, porous structures) for applications in biotechnology, nutrition, tool engineering etc.,
- Ion-beam analysis for the chemical analysis of thin films or geological samples,
- Accelerator mass spectrometry of non-radiocarbon samples

The semiconductor processing on wafer level requires an effective routine for large volume processing. For that reason, we are operating - at the 6 MV, 3 MV accelerators and at the 500 kV ion implanter – in total four semi-automatic wafer handling systems for effective processing of all kinds of semiconductor wafers (not only SEMI standard silicon wafers). For more information, the corresponding IBC webpages or the instrument scientists should be contacted. Figure 2 shows some process stations used for ion beam services.

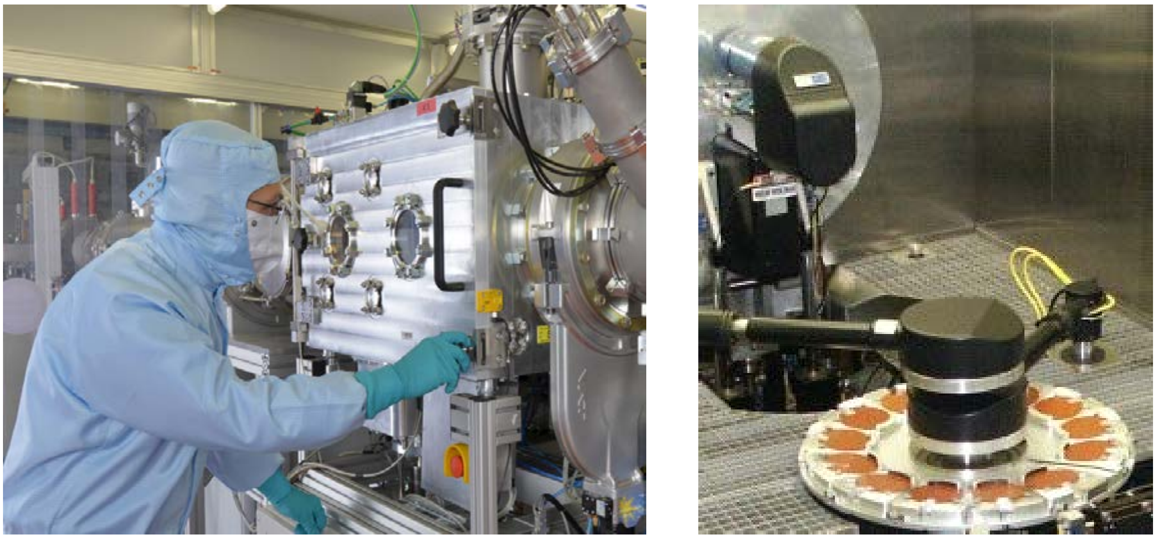

Figure 2: Implantation chamber (part of semi-automatic wafer handler system, ∅ 200 mm wafer capability) at the 6 MV tandem (left), and wafer wheel (max. wafer size 150 mm) of the NV-10 end-station used for high-throughput implantations at the 3 MV tandem (right).

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