

Technical description of research modular platform Nanolab

Resource center «Physical Methods of Surface Investigations»

2016

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Common description

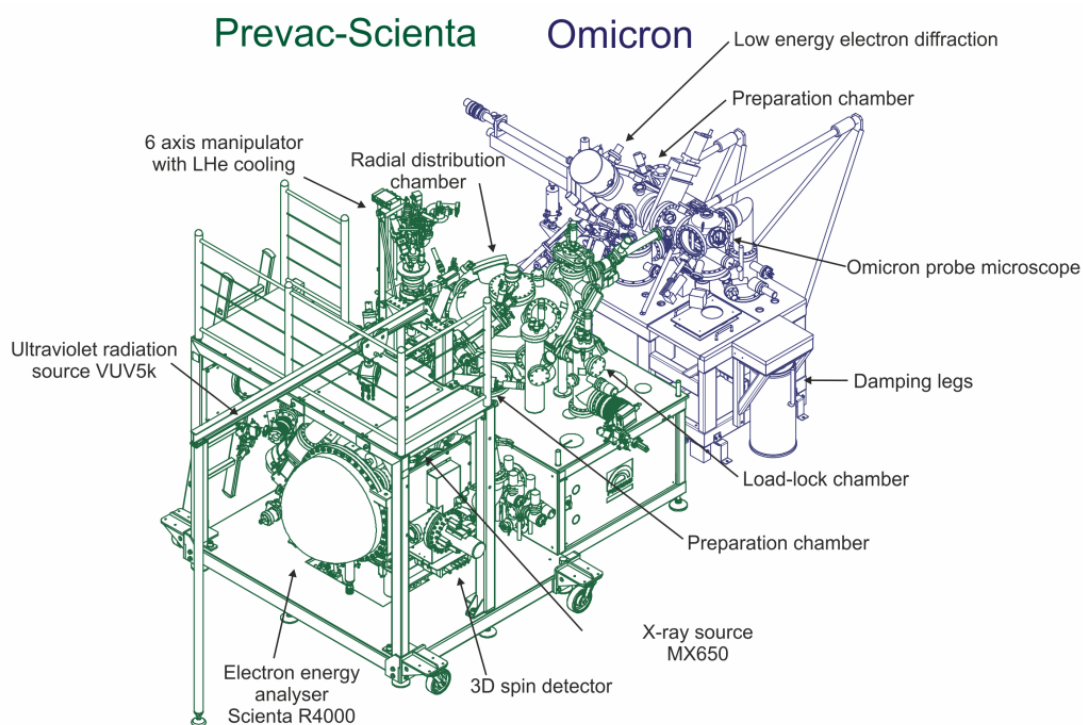


Figure 1: General scheme of research modular platform Nanolab.

Research modular platform Nanolab consists of 2 modules: Prevac - VG Scienta and Omicron (see fig. 1 and 2). These modules are connected for transferring of samples between them without disruption of ultrahigh vacuum conditions and are independent experimental stations. Thus, it is possible to make consequent measurements of one sample in modules Prevac - VG Scienta and Omicron or make parallel measurements of 2 samples simultaneously.

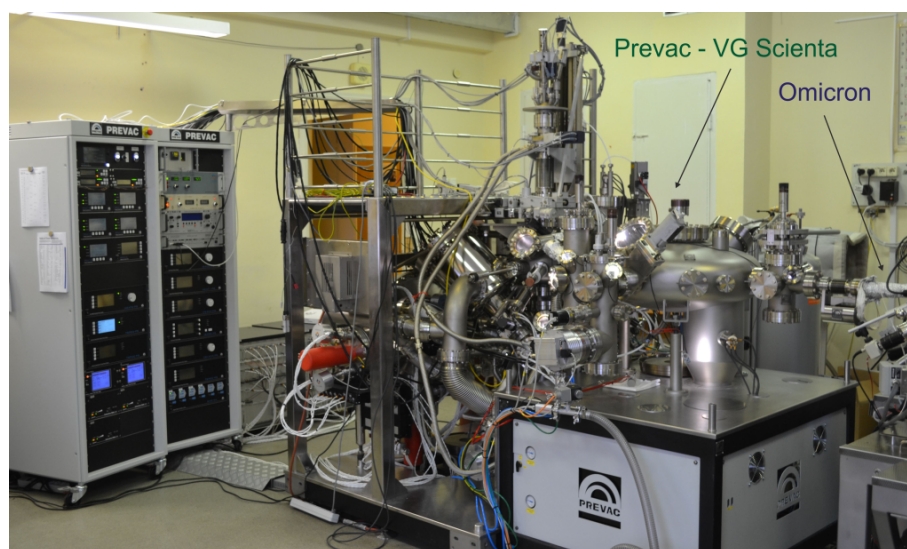


Figure 2: Common view of research modular platform Nanolab.

Module Prevac - VG Scienta

Common description

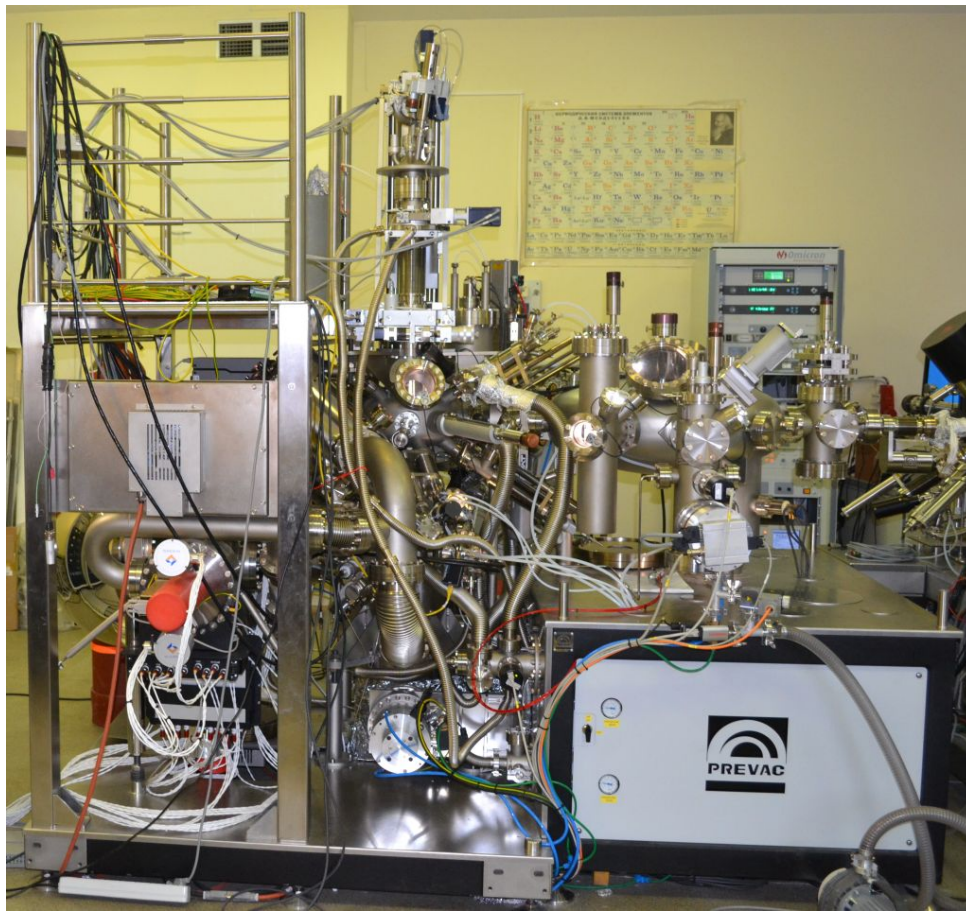


Figure 3: Module Prevac - VG Scienta.

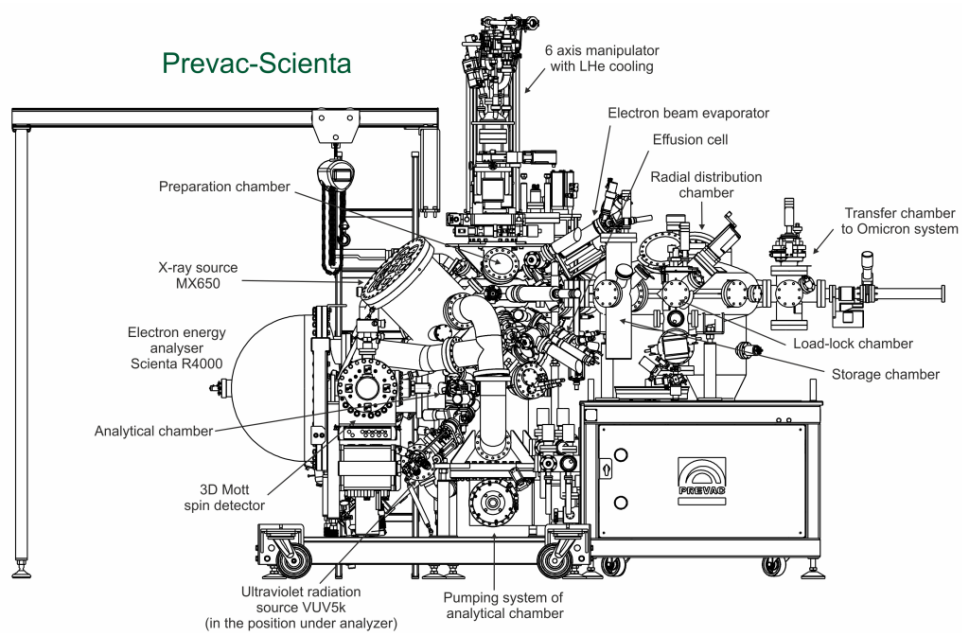


Figure 4: Drawing of module Prevac - VG Scienta.

Following methods are available in module Prevac - VG Scienta:

1. Photoelectron spectroscopy (PES) of core levels (source: VG Scienta MX 650, see section «Analytical chamber»)
2. Spin and Angle Resolved Photoelectron Spectroscopy (SARPES) of valence band (analyzer: VG Scienta R4000 with 3D spin-detector, source: VG Scienta VUV 5k see section «Analytical chamber»)
3. Low Energy Electron Diffraction (LEED) (diffractometer: OCI BDL800IR, see section «Preparation chamber»)
4. Auger Electron Spectroscopy (AES) (optics of diffractometer OCI BDL 800IR is used, see section «Preparation chamber»)

Available information about objects::

1. Electron and spin structure of valence band and core levels

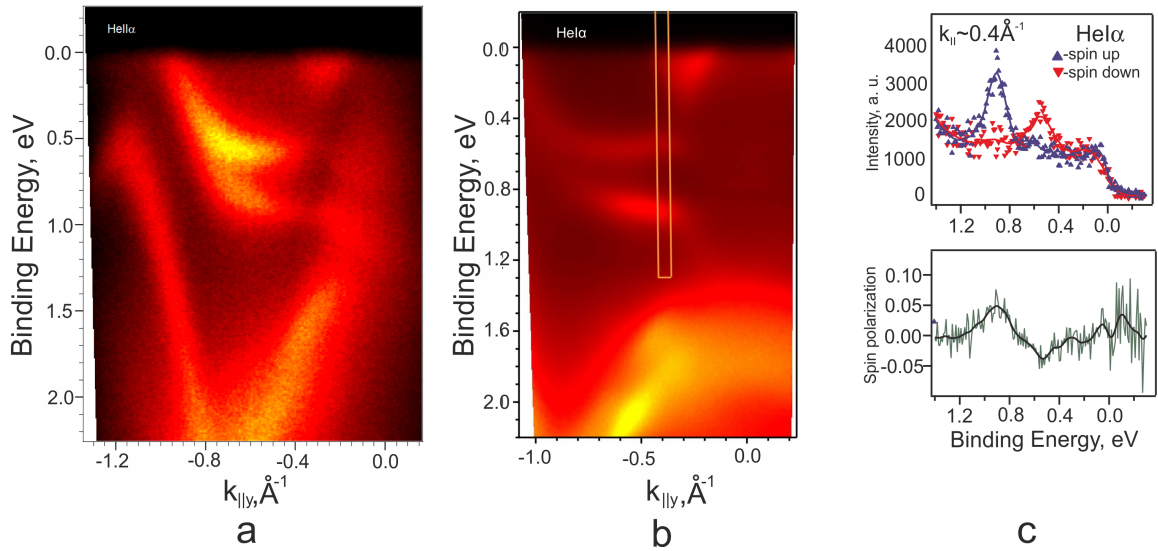


Figure 5: Spin-resolved photoelectron spectra of system 1 ML Au / W(110) in $\bar{\Gamma}\bar{S}$ -direction of Brillouin zone: (a) photon energy - 40.8 eV (line HeI α), (b) photon energy - 21.2 eV (line HeII α), (c) spin-resolved spectra and its spin polarization, photon energy -21.2 eV (line HeI α)

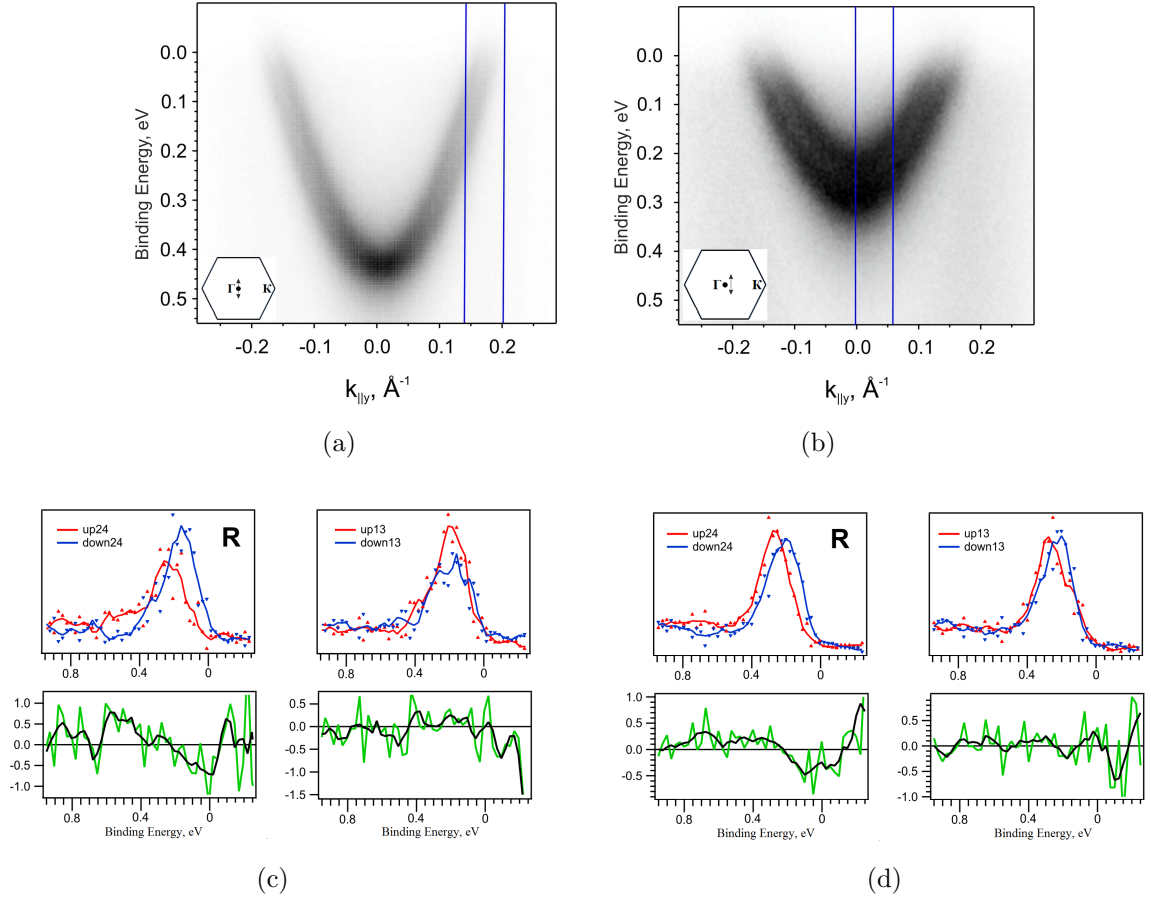


Figure 6: Shockley surface states at Au(111): (a) photon energy - 21.2 eV (line HeI α), $E_p = 5$ eV, lens mode - 14°, slit - 300 (0.2 mm), (b) photon energy - 21.2 eV (line HeI α), $E_p = 5$ eV, lens mode - 14°, slit - 300 (0.2 mm), polar angle - 3°, (c) spin-resolved spectra for $k_{||x} = 0$, $k_{||y} \sim 0.17 \text{\AA}^{-1}$, photon energy - 21.2 eV (line HeI α), $E_p = 10$ eV, lens mode - 14°, slit - 500 (0.5 mm), "white" detector, aperture #3, tilt angle - 3.5°, (d) spin-resolved spectra for $k_{||x} = 0.15 \text{\AA}^{-1}$, $k_{||y} \sim 0.03 \text{\AA}^{-1}$, photon energy - 21.2 eV (line HeI α), $E_p = 10$ eV, lens mode - 14°, slit - 400 (0.3 mm), "black" detector, aperture #3, polar angle - 3.5°.

2. Crystal structure

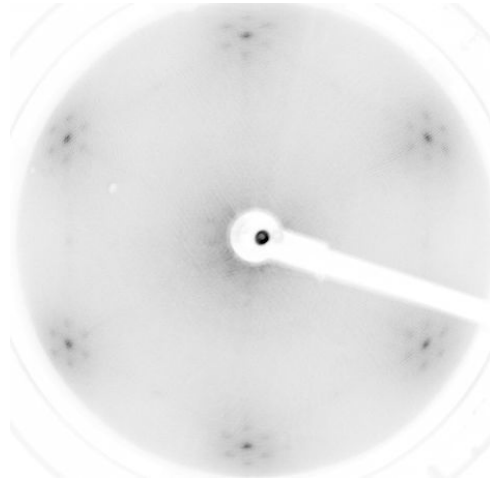


Figure 7: LEED image of structure graphene/Au/Co/W110. Beam energy - 110 eV.

3. Chemical shifts

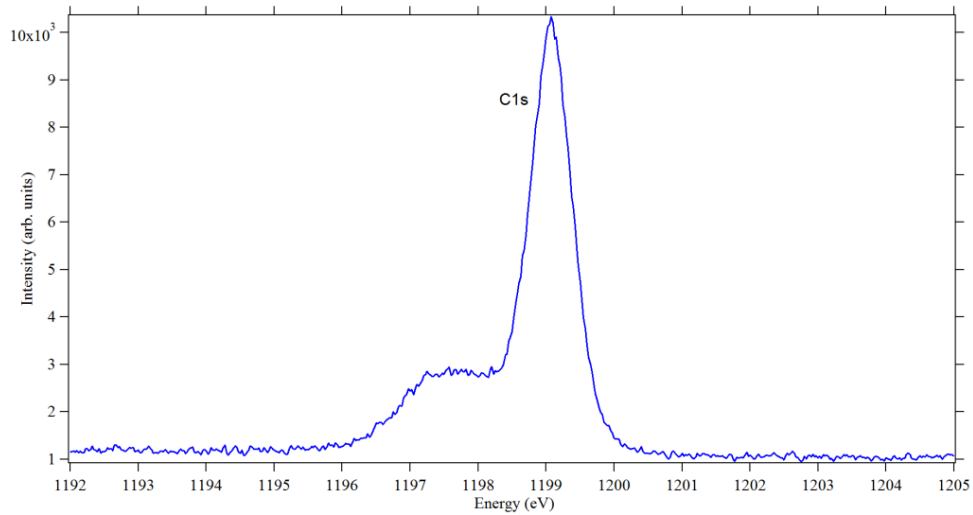


Figure 8: Carbon in different chemical states in compound SiC(1000).

Analytical chamber Prevac - VG Scienta

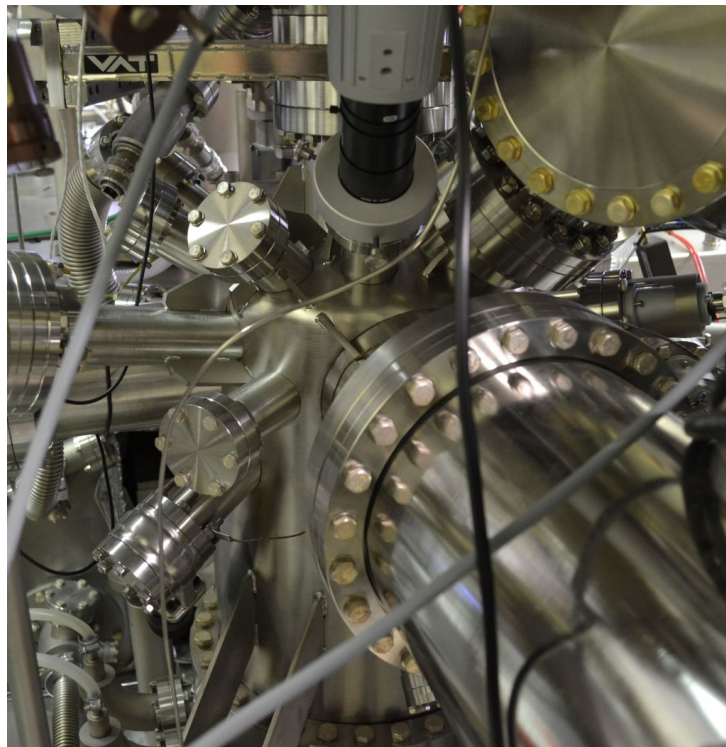


Figure 9: Analytical chamber of module Prevac - VG Scienta.

Experimental station consists of four chambers, which have independent pump-down systems (see fig. 31): analytical chamber (see fig. 9), preparation chamber (see fig. 11), radial distribution chamber (see fig. 14) and load lock chamber (see fig. 12). Two another chambers are mounted to radial distribution chamber: storage chamber (see fig. 13(a)) and PTS adapters reorientation chamber (see fig. 13(b)). Base pressure in analytical chamber is $1 - 2 \cdot 10^{-10}$ mbar.

Material of analytical chamber: μ -metal. Analytical chamber contains following equipment: (see fig. 10):

1. Energy analyzer VG Scienta R4000 WAL-01.1 XPS/UPS/ARPES with 3D spin-detector
2. Monochromate X-ray source VG Scienta MX-650: source SAX-100 K_{α} -Al, monochromator XM-780
3. Digital camera
4. High intensity narrow bandwidth UV-source VG Scienta VUV 5k with extensible capillar
5. Ion source Prevac FS40A1 for charge compensation (with shield)¹
6. Laser pointer
7. Raster ion source Prevac IS40E1²

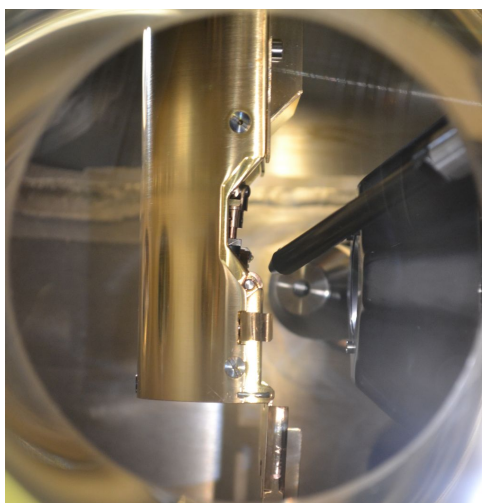


Figure 10: Inside analytical chamber of module Prevac - VG Scienta.

Preparation chamber Prevac - VG Scienta

Material: stainless steel. Preparation chamber (see fig. 11) contains following equipment:

1. Thickness monitor Prevac TM13³
2. Effusion cell Prevac EF 40C1⁴
3. Electron beam evaporator Prevac EBV 40A1⁵

¹see <http://www.prevac.eu/en/2,offer/37,instruments/124,flood-source-40a1.html>

²see <http://www.prevac.eu/Media/catalog/file/en/6/rys-tech-ion-source-is40e1.pdf>

³see <http://www.prevac.eu/en/news,10/27,new-thickness-monitor-controller-tmc-13.html>

⁴see <http://www.prevac.eu/en/2,offer/37,instruments/126,effusion-cell-ef-40c1.html>

⁵see <http://www.prevac.eu/en/2,offer/37,instruments/125,electron-beam-evaporator-ebv-40a1.html>

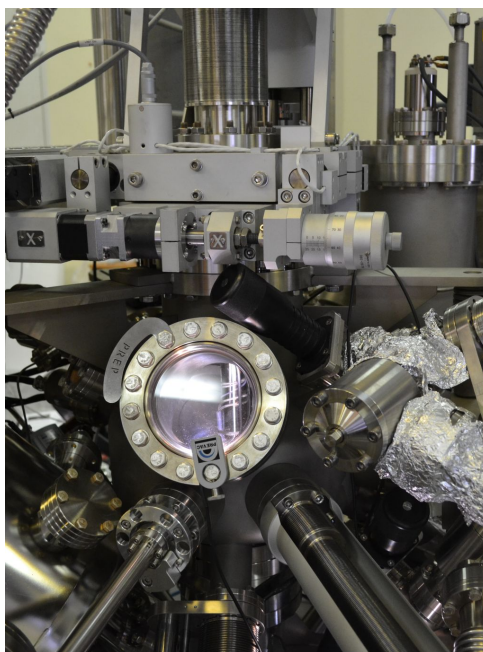


Figure 11: Preparation chamber of module Prevac - VG Scienta.

4. Diffractometer OCI BDL800IR with optics for Auger-electron spectroscopy⁶
5. Ion source Prevac IS 40C1⁷
6. 6-axes motorized⁸ manipulator Prevac with possibility of cooling by liquid helium (common manipulator with analytical chamber)
7. Gas analyzer SRS RGA100⁹
8. Flashing machine (temperature of heating - up to 2000 °C)
9. System of transferring of samples without disruption of ultrahigh vacuum conditions

⁶see http://www.ocivm.com/spectrometer_bdl800ir.html

⁷see <http://www.prevac.eu/en/2,offer/37,instruments/123,electron-source-40c1.html>

⁸see <http://www.prevac.eu/en/2,offer/44,motor-controllers-for-simple-transferring-system/87,smcd-10.html>

⁹see <http://www.thinksrs.com/products/RGA.htm>

Load-lock chamber Prevac - VG Scienta

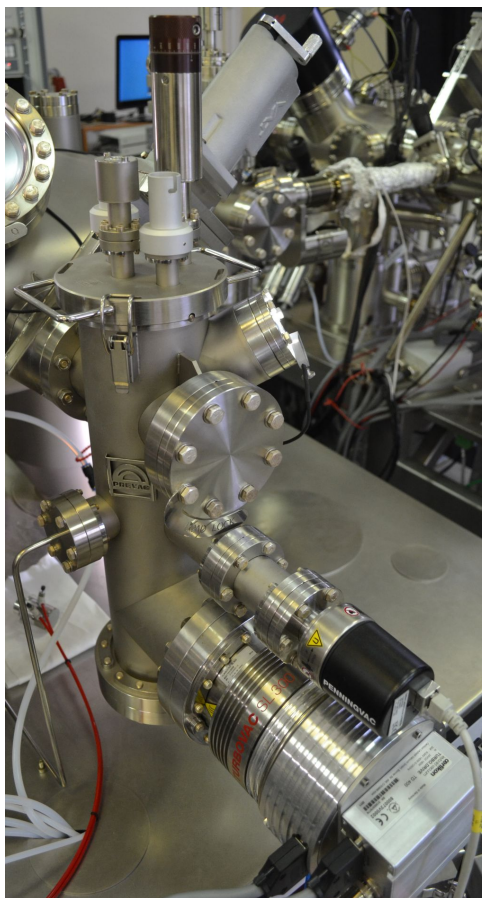


Figure 12: Load-lock of module Prevac - VG Scienta.

Load-lock chamber (see fig. 12) is part of system, in which direct input of samples from atmosphere and output of them after measuring are performed. Input is realized by special section, which is segregated from experimental station and is mounted on it with viton ring. It is possible to take out from chamber up to 4 PTS/OMC adapters (see section «Requirements for samples. Sample holders. Transfer of samples between modules»). Load-lock is not ultrahighvacuum chamber, but pump-down system allows to get pressure near $1 \cdot 10^{-8}$ mbar.

Storage chamber Prevac - VG Scienta

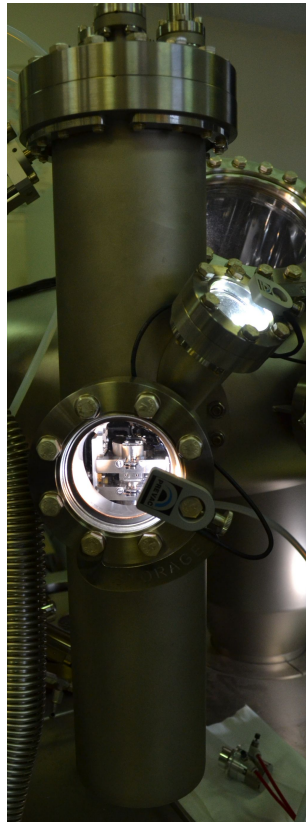
Storage chamber (see fig. 13(a)) is used for allocation of samples between experiments in ultrahighvacuum conditions. It is possible to mount up to 10 PTS/OMC adapters (see section «Requirements for samples. Sample holders. Transfer of samples between modules»).

Sample reorientation chamber Prevac - VG Scienta

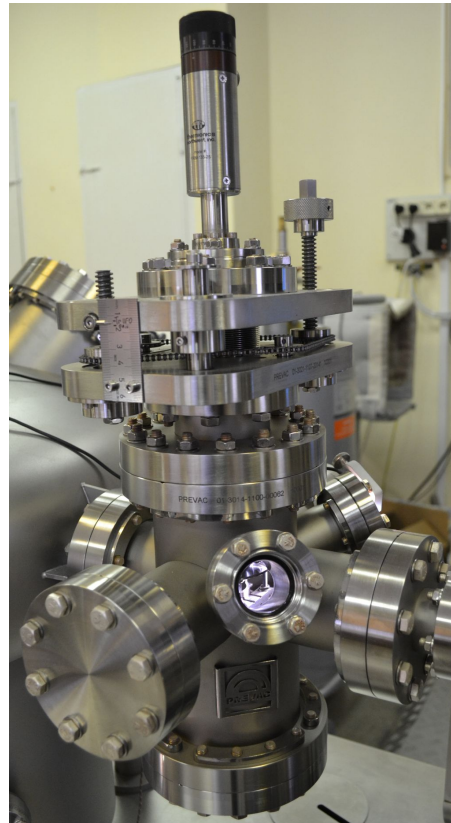
Sample reorientation chamber (see fig. 13(b)) is located between radial distribution chamber of module Prevac - VG Scienta and analytical chamber of module Omicron and is designated for temporary allocation of sample holders during transferring of samples between modules.

Sample reorientation chamber contains following equipment:

1. ultrahigh vacuum chamber
2. 2-axes manipulator
3. windows and blind flanges
4. metallic sylphon for vibroisolation
5. manual valve DN VAT DN40CF



(a)



(b)

Figure 13: (a) storage chamber, (b) sample reorientation chamber

Radial distribution chamber of module Prevac - VG Scienta

Radial distribution chamber (see fig. 14) is designated for transferring of samples between load-lock chamber, storage chamber, radial distribution chamber and preparation chamber.

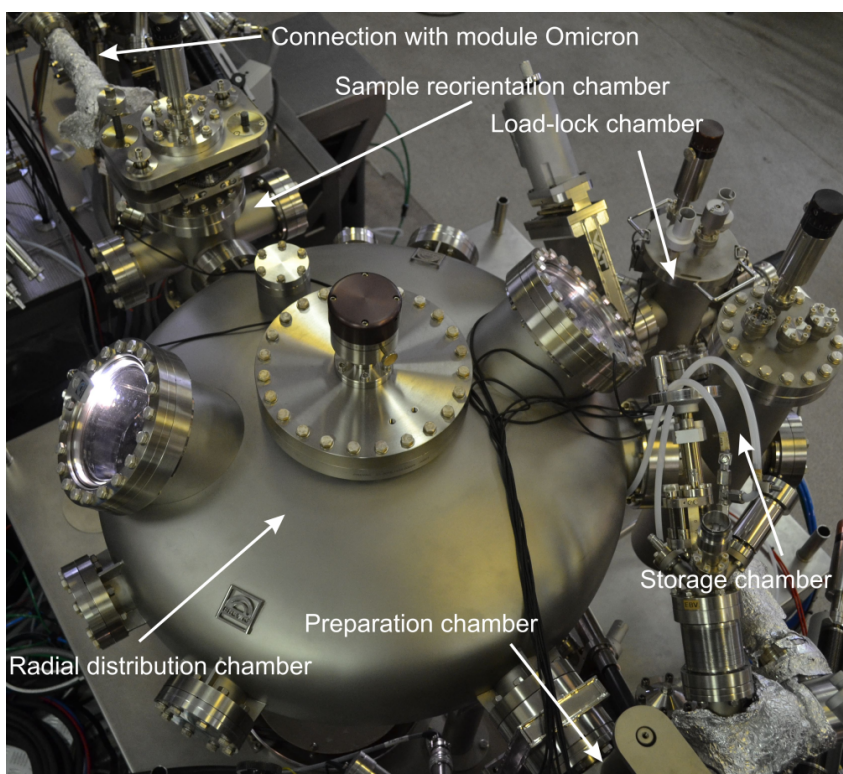


Figure 14: Radial distribution chamber of module Prevac - VG Scienta with sample reorientation chamber and load-lock chamber.

Module Omicron

Common description

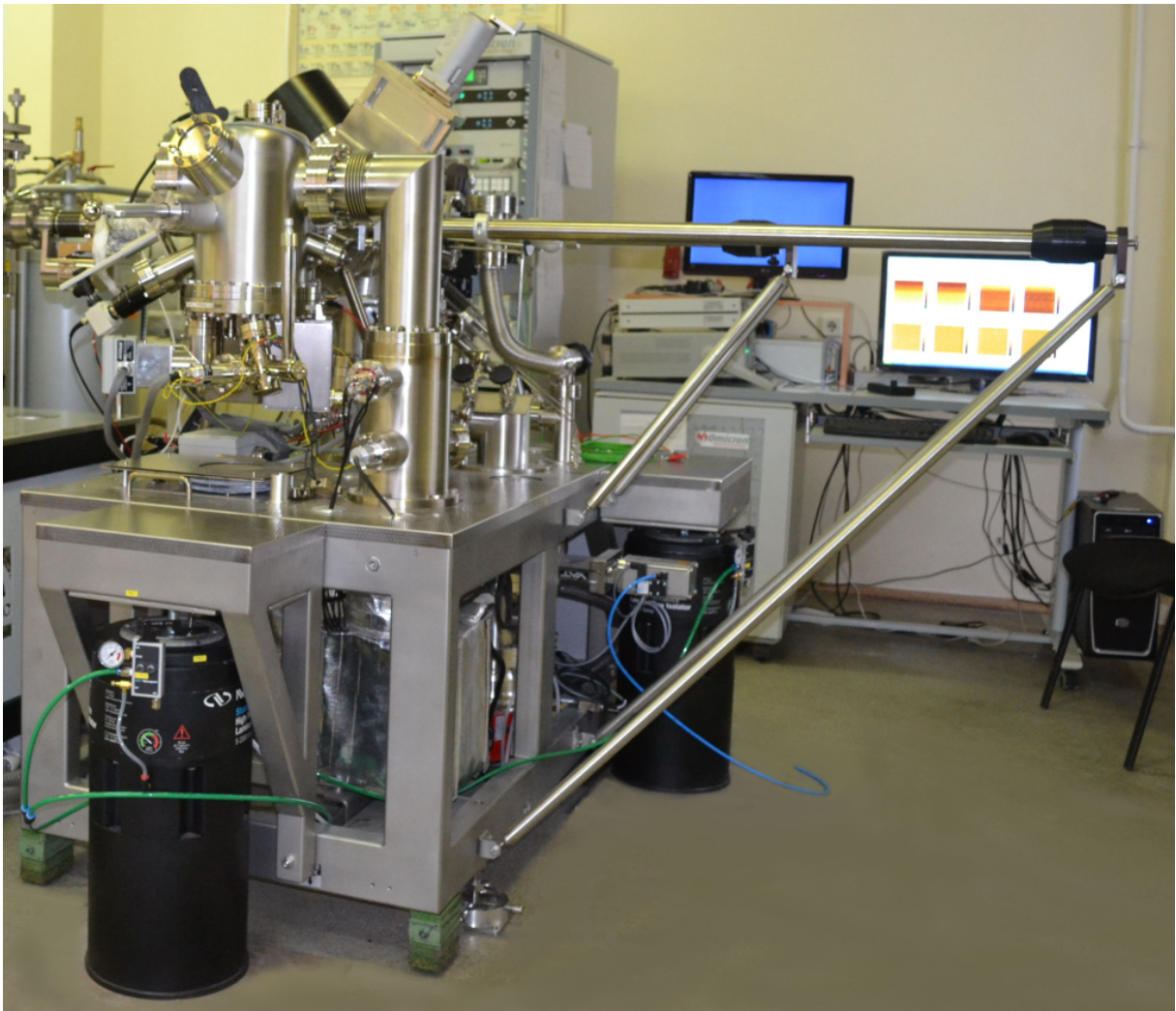


Figure 15: Module Omicron.

Following methods are available in module Omicron:

1. Scanning tunneling microscopy (STM) and Scanning tunneling spectroscopy (STS): obtaining images of surface with atomic resolution, energy spectra of occupied and free states with high lateral resolution.
2. Atomic force microscopy (AFM) (contact and non-contact modes): obtaining images of surface of samples (including non-conductive samples)
3. Low-energy Electron Diffraction (LEED)
4. Auger electron spectroscopy (AES)

Station is attached to nitrogen gas line with possibility of independent venting of chamber of recharging and each source without violation of vacuum conditions in

preparation chamber and analytical chamber. Ion source is attached to pumpable gas line.

Available information about objects:

1. information about topography of surface with atomic resolution

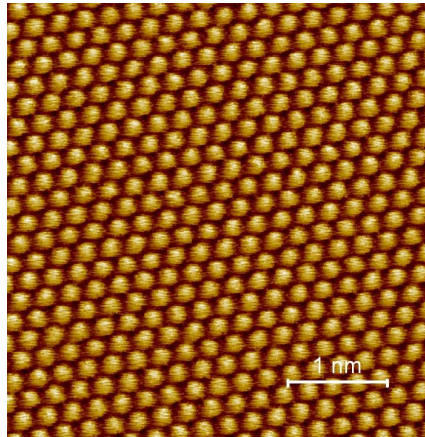


Figure 16: STM-image (current channel, W-tip), HOPG room temperature. Parameters of scanning: current channel $I_t = 1.2$ nA, sample voltage $V_s = -0.2$ V, loop gain - 1%, scanning speed - 39 nm/s.

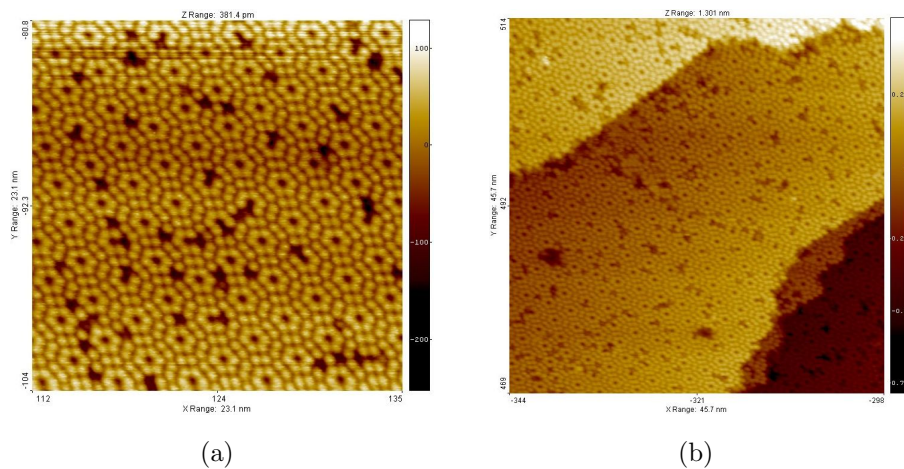


Figure 17: STM-image Si(111) 7×7 : (a) obtained at room temperature, (b) temperature 400 K. Parameters of scanning: scanning current $I_t = 0.3$ nA, sample voltage $V_s = -1.2$ V, loop gain - 6%, scanning speed - 29 nm/s.

2. information about crystallographic structure and orientation of surface

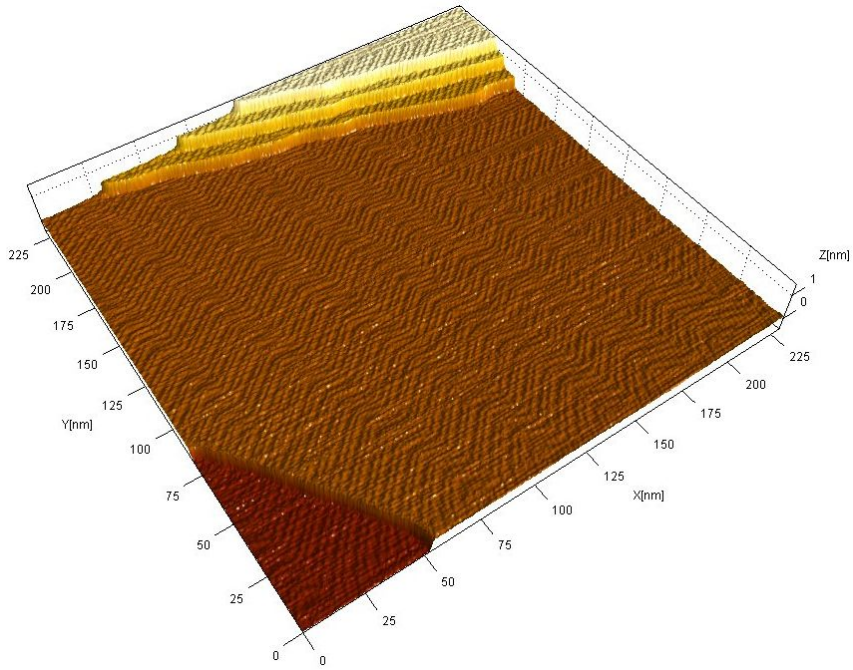


Figure 18: STM-image Au(111) $22 \times \sqrt{3}$ ¹¹ obtained at room temperature. Parameters of scanning: tunnelling current $I_t = 0.3$ nA, sample voltage $V_s = -0.3$ V, loop gain - 1.5%, scanning speed - 250 nm/s.

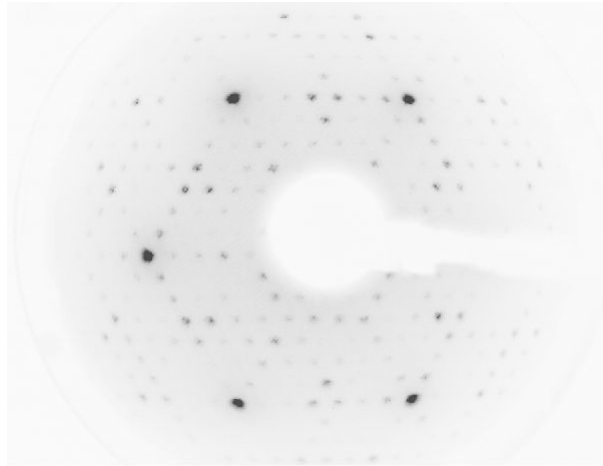


Figure 19: LEED image Si(111)7×7. Beam energy - 50 eV.

Analytical chamber: design, technics, measurements

Experimental station consists of three chambers, which have independent pump-down systems: analytical chamber (see fig. 21), preparation chamber (see fig. 23) and load-lock (see fig. 24). Base pressure in analytical chamber is better than $1 \cdot 10^{-10}$ mbar.

Analytical chamber contains following equipment: (see fig. 22):

¹¹herringbone structure - see [Felix Hanke and Jonas Björk - Phys. Rev. B 87, 235422](#)

1. scanning probe microscope VT AFM XA 50/500¹²
2. storage for placing 12 sample holders and probes
3. device for preparing cantilevers for SPM
4. digital macrocamera

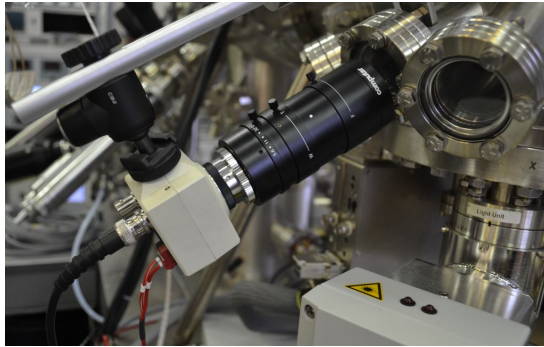


Figure 20: Digital macrocamera.

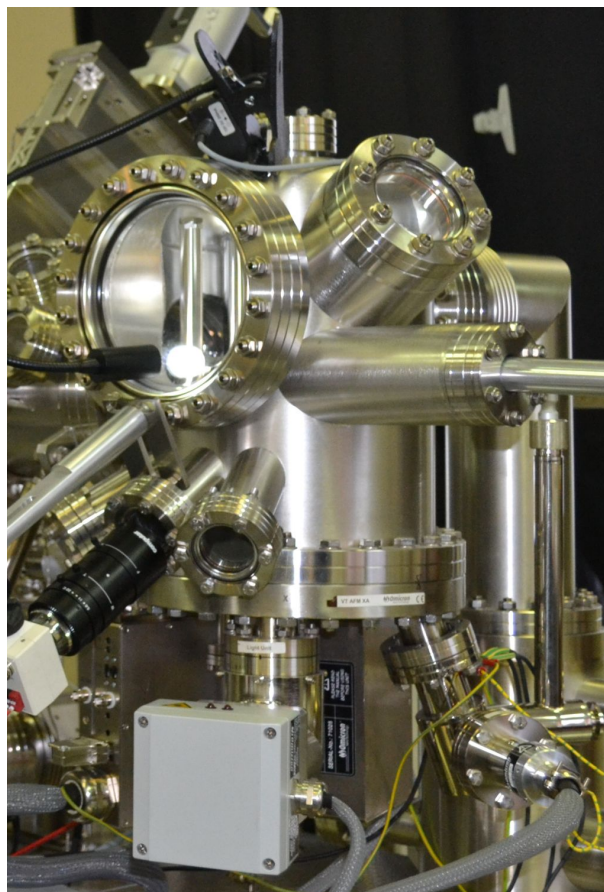


Figure 21: Analytical chamber Omicron.

¹²see <http://www.scientaomicron.com/en/products/variable-temperature-spm/variants#page224>

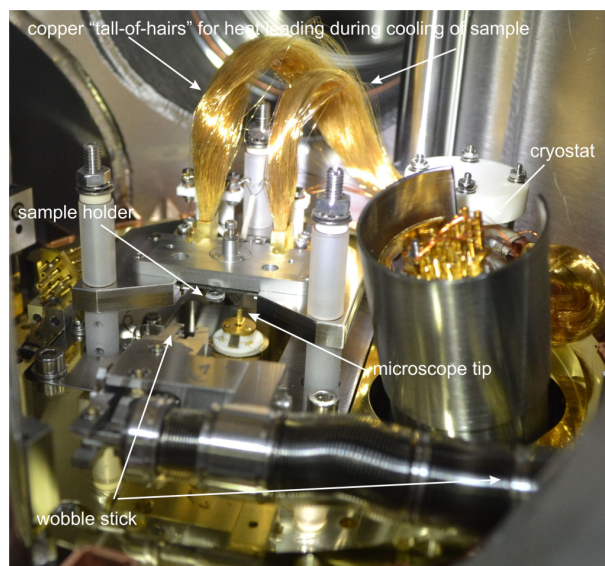


Figure 22: Inside analytical chamber Omicron.

Preparation chamber

Preparation chamber contains following equipment:

1. four grid low-energy electron diffractometer Omicron SPECTALEED with built-in Auger electron spectrometer¹³
2. four axis manipulator VG Scienta with heating up to 1050 K due to heating radiation and possibility of heating conductive samples by direct current
3. 3 positions of installing sources for precise (up to 1 monolayer) evaporation of thin films: by thermal heating (WEZ¹⁴) and by electron bombardment (EFM 3¹⁵)
4. ion gun ISE 5¹⁶ for etching of surface of samples (beam energy: from 0.3 to 5 keV, beam current: up to 80 μA when 5 keV and 25 μA when 0.5 keV, spot size: 10 mm when 5 keV, 15 mm when 1 keV)

Load-lock chamber

Load-lock chamber is part of system, where samples are installed to experimental station from atmosphere and their unloading after measurements. Input is realized through a flange CF63, holder with sample is fixed at transfer for further transferring to preparation chamber. It is possible to put (or unload) one sample holder, which is located at transfer, during on cycle. Load-lock chamber is not ultrahighvacuum chamber, but pump-down system allows to get pressure near $1 \cdot 10^{-8}$ mbar.

¹³see <http://www.scientaomicron.com/en/products/spectaleed-/instrument-concept>

¹⁴see <http://www.scientaomicron.com/en/products/wez-/instrument-concept>

¹⁵see <http://www.scientaomicron.com/en/products/efm-3-/instrument-concept>

¹⁶see <http://www.scientaomicron.com/en/products/ise-5-/instrument-concept>

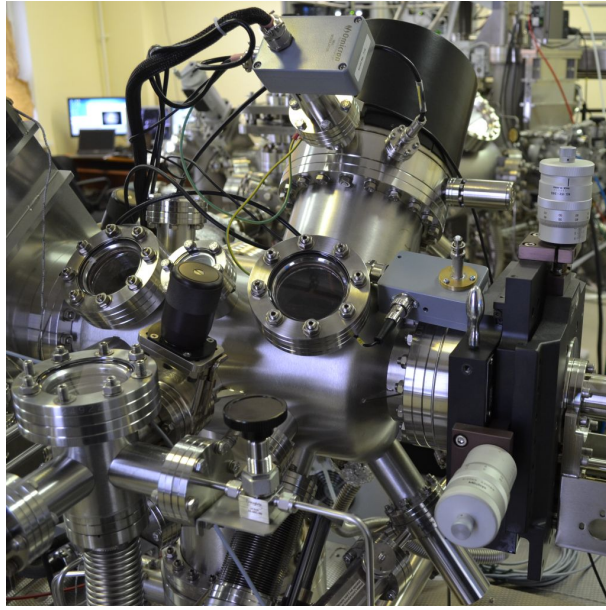


Figure 23: Preparation chamber Omicron (left bottom - load-lock chamber).

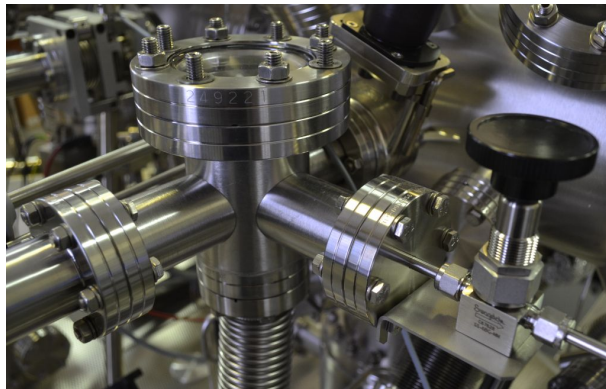


Figure 24: Load-lock chamber.

Requirements for samples. Sample holders. Transfer of samples between modules.

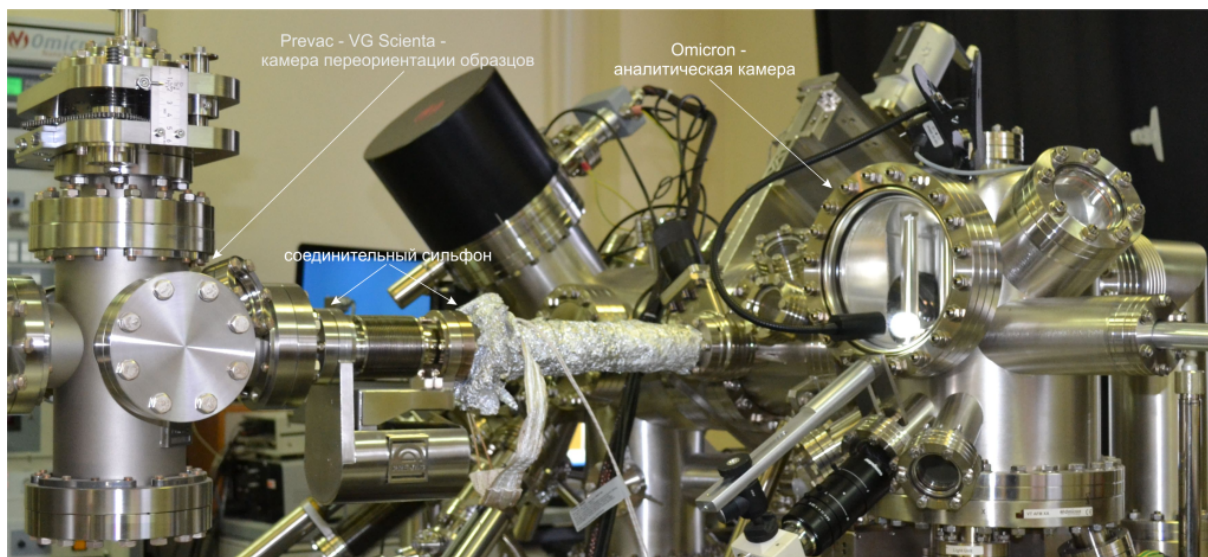


Figure 25: Connecting sylphon between modules Prevac - VG Scienta and Omicron.

Samples are placed directly into standard sample holders Omicron (Omicron-type plates or OMC¹⁷ - see fig. 26(a)). Sample size: lower than 11x11 mm. with fastening elements (see fig. 26(b)), thickness - up to 3 mm. It is possible to measure solid-state samples, which don't disrupt ultrahighvacuum conditions during experiments. Assembling of samples to sample holders can be done beforehand by users (sample holders can be done according to drawing - see fig. 27) and by staff of resource center directly before experiment.

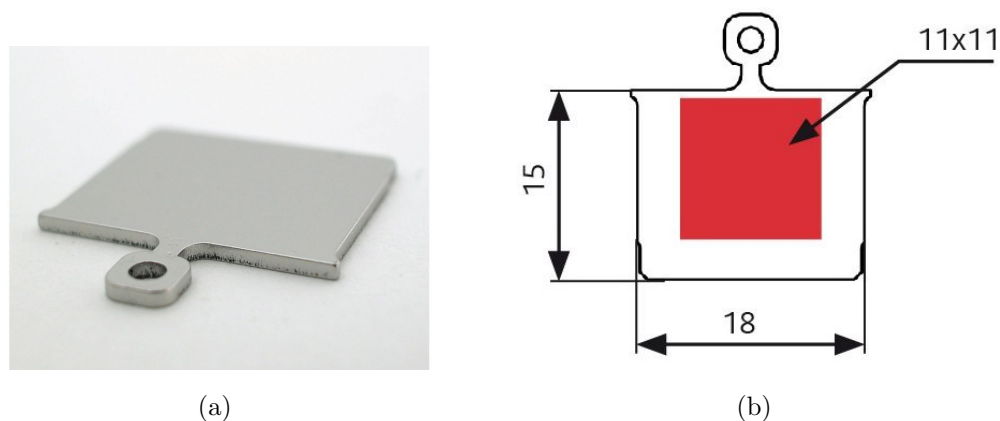


Figure 26: Standard sample holder Omicron (standard sample plate): (a) common view, (b) work area (marked by red)

¹⁷see <http://www.scientaomicron.com/en/products/multiprobe-systems-general-information/further-details#page279>

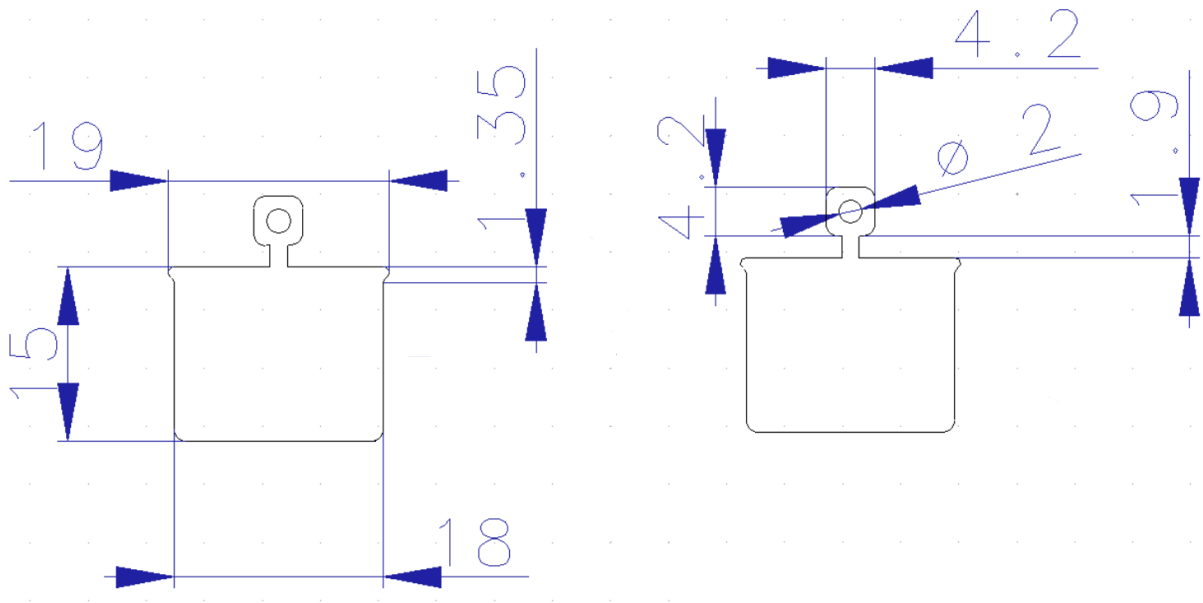


Figure 27: Scheme of standard sample holder Omicron (OMC).

Direct current heating in module Omicron is realized at special sample holder (see fig. 28).

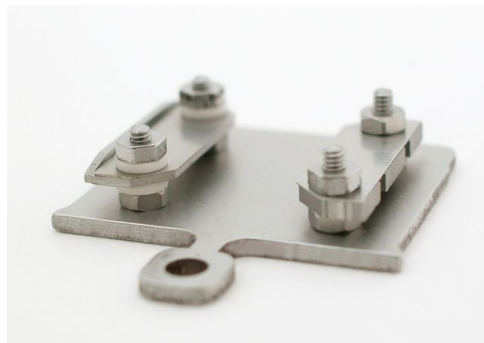


Figure 28: Sample holder for direct current heating (SP DC).

Also there are sample holder for holding crystals and sample holder with hole for minimization of losses of heat during heating a sample (see fig. 29).

At fig. 30 sample holder with mounted monocrystal is shown.

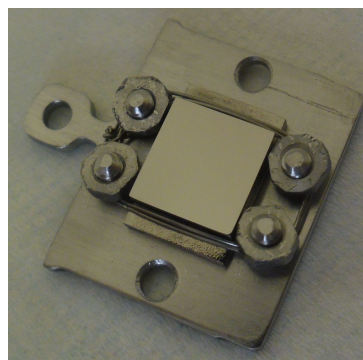


Figure 30: Sample holder with mounted monocrystal.

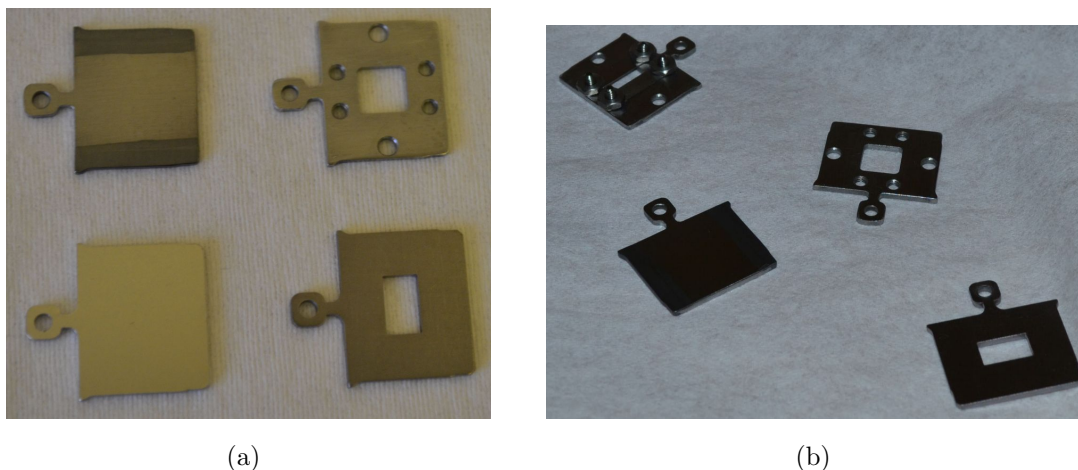


Figure 29: Sample holder: (a) top view: on the left - standard sample holder, top right - sample holder for mounting crystals with hole, (b) common view.

In operation with module Omicron it is possible to put 1 sample holder through load-lock chamber of module Omicron. Analytical chamber of module Omicron contains storage, which can hold 12 sample holders.

In operation with module Prevac - VG Scienta adapters Prevac of PTS/OMC type are used, each can hold 1 or 2 sample holders OMC. It is possible to enter to load-lock chamber of module Prevac - VG Scienta up to 4 adapters PTS/OMC (maximal quantity of sample holders OMC, which can be mounted in these 4 adapters PTS/OMC, depend on sizes of samples). After getting necessary vacuum conditions in load-lock chamber, it is possible to move adapters PTS/OMC to storage chamber and to preparation chamber of module Prevac - VG Scienta through UFO-chamber. During movement to storage chamber is it possible to mount adapter at storage area (it is possible to mount 10 adapters PTS/OMC at storage are simultaneously). During movement to preparation chamber 1 sample holder OMC is taken out from adapter PTS/OMC by manual wobble-stick and is moved to manipulator Prevac - VG Scienta, which can be moved further to analytical chamber of module Prevac - VG Scienta.

It is possible to move samples without disruption of vacuum conditions between analytical chamber of module Omicron and radial distribution chamber of module Prevac - VG Scienta. During movement from module Prevac - VG Scienta to module Omicron one adapter PTS/OMC is moved to ground, which is located in reorientation chamber of module Prevac - VG Scienta. Then it is possible to move 1 sample holder OMC to module Omicron by magnet linear manipulator.

Figure 31 shows principal vacuum scheme of platform Nanolab: vacuum chambers, where direct work with OMC sample holders is done, are marked with red, vacuum chambers, where moving of the samples is done in adapters PTS/OMC, valves, which allow to disconnect parts of experimental station, are marked with blue.

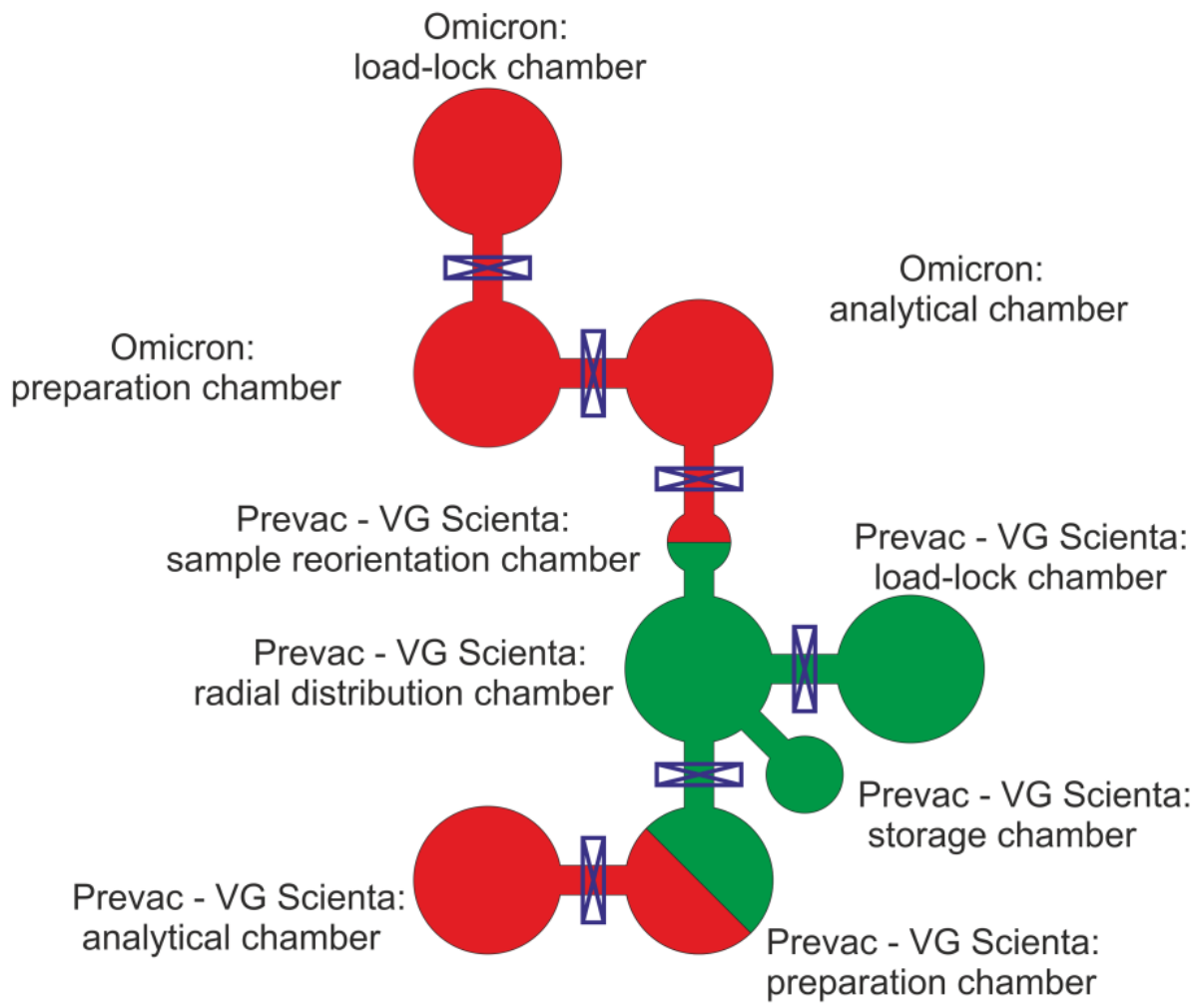


Figure 31: Vacuum scheme of experimental station Nanolab.