IKBFU Research and Educational Centre

«Functional Nanomaterials»

Опыт создания и эксплуатации в БФУ комплекса на основе низкоэнергетичного ускорителя. Материалы и инструменты для перспективных нейтронных и синхротронных исследований.

Работа выполнена в БФУ им. И. Канта в рамках соглашения № 14.584.21.0028 с Минобрнауки РФ
Уникальный идентификатор проекта RFMEFI58417X0031

Александр Гойхман

Immanuel Kant Baltic Federal University

Functional Nanomaterials
RESEARCH AND EDUCATION CENTER

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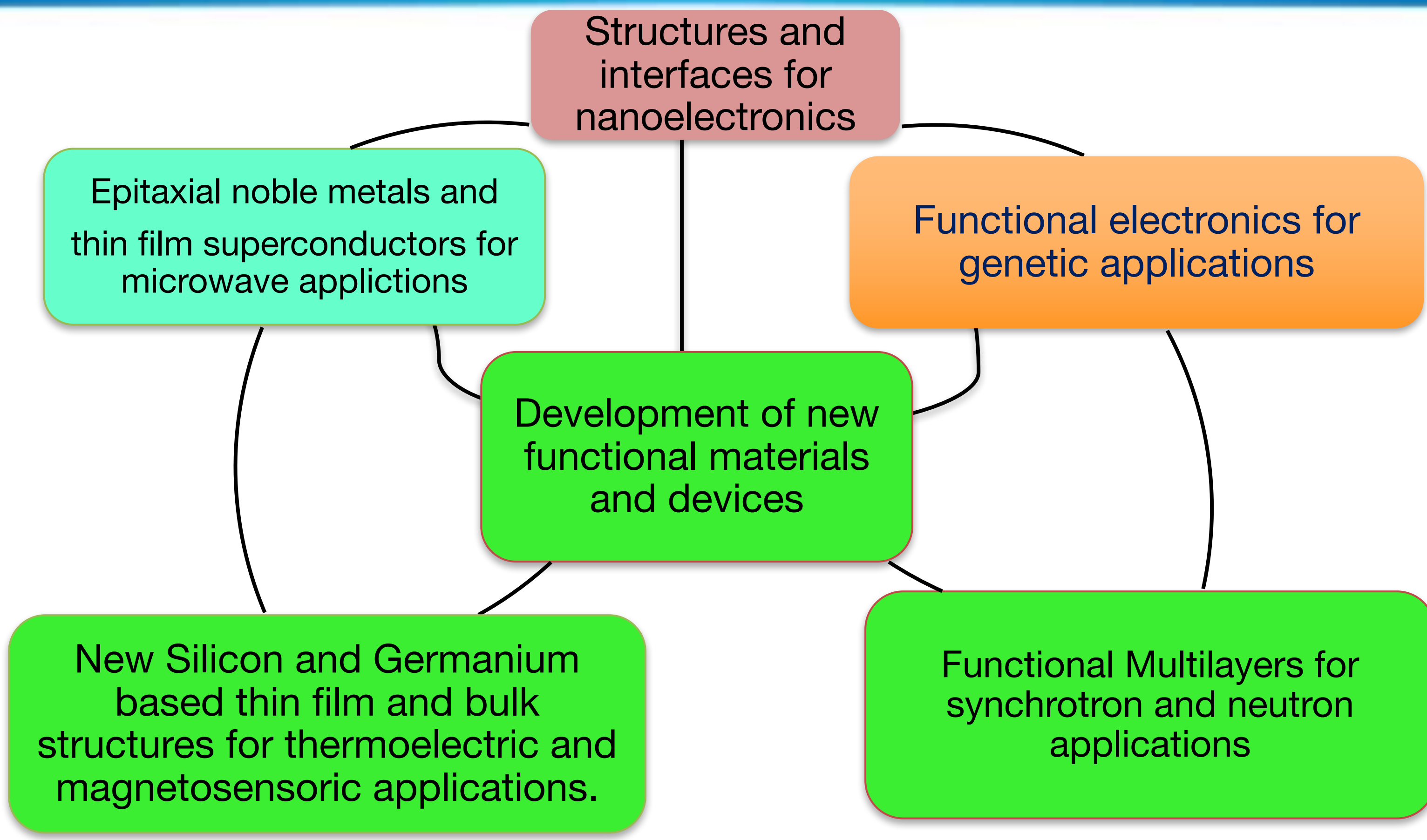
«Functional Nanomaterials»

20 persons, responsible A. Goikhman

- 5 Ph. D students (O. Yurkevich, O. Dikaya, U. Koneva, D. Serebrennikov, A. Grunin)
- 3 Master Students (A. Kozlov, E. Maznitsyna, A. Shapilov)
- 4 Postdocs and researchers: E. Klementyev, P. Shvets, K. Maksimova, A. Vinichenko
- 7 Engineers: (P. Prokopovich, D. Efimov, V. Kolesniskiy, A. Dolgoborodov, V. Molchanov, E. Severin, V. Fedotov

REC FN Scientific Program



Totally 40 publications with REC since 2014 in Scopus/WoS with > 100 citations (for this papers)

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E. Klementyev

Strongly Correlated Systems Lab

- High pressure cells
- Models and methods for electronic systems calc

K. Maksimova

Magnetron+ UHV **Pulsed Laser Deposition** SVTA(2010)

Atomic Layer Deposition SVTA(2011)

AFM/MFM UHV JEOL(2008)

ToF SIMS Oxford Instruments (2011)

Ion Beam Deposition (2008)

Be Magnetron Sputtering

RF/DC Large Area Magnetron Sputtering (2013)

AES + HR-SEM JEOL(2009)

SEM +EDS JEOL(2009)

TEM JEOL + sample preparation

XRD/XRR with GISAXS+GID Bruker D8 (2011)

Raman-AFM complex Horiba LabRAM+AistNT (2012)

UV - IR Sph Shimadzu (2010)

D. Efimov

Ion Beam sector

HVEE Van der Graff accelerator (RBS, ion implantation)

He+, protons, liquid and solid ion sources

Engineering group

- Equipment support
- Drawings
- New Facilities Development

P. Prokopovich

Secretary group

- Project time management
- Applications
- Trips organization

I. Smirnova

2-MeV Ion Acceleretor

Технические характеристики системы: Диапазон масс	В ±150 321 а.е.м
Диапазон энергии	200-2000 КэВ для одного вида ионов
Ток пучка диапазон: 800-2000KV 400-800KV 200-400KV	4He+; 11B+; 16O+; 28Si+; 31P+; 40Ar+; 75As+ ≈150μA; ≈40μA; ≈35μA; ≈40μA; ≈40μA; ≈200μA; ≈40μA примерно 70% от указанных выше значений примерно 50% от указанных выше значений
Пульсация тока пучка	± 10%
Стабильность напряжения на клеммах	Пульсация ±2 KV Напрааление ±2 KV

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2-MeV Ion Acceleretor

unique ion-beam research cluster, based on the new HVEE Ion Accelerator

2-MeV Ion Acceleretor

Области применений:

- Ion-beam analysis (Rutherford and non-Rutherford, PIXE, PIGE)
- Ионная имплантация для силовой электроники
- Испытания радиационной стойкости материалов и устройств

- Исследования и модификация биологических объектов
- Наработка специальных короткоживущих изотопов (F18), время жизни ~ несколько часов
- Эксперименты по созданию нейтронных пучков ($p+^{7}Li=n+^{7}Be$):

энергии нейтронов сотник КэВ, порог реакции 1.88 MeV

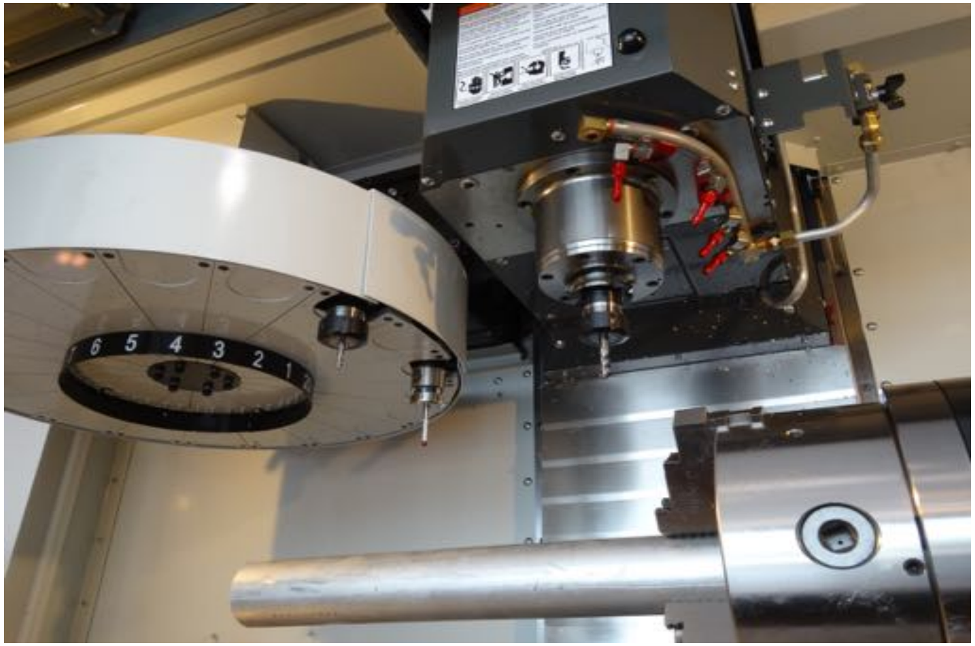
Engineering facilities of REC FN

- Precious metal machining
- Accuracy - 1 um
- Maximum details sizes – 1 m

- milling, lathing
- Argon welding stage

- Development of vacuum chambers and setups
- CAD models development
- Calculation of strength properties

CNC vertical milling machine



Types of processed materials

Steel

Non-ferrous metals

Polymeric materials

Main parameters and features:

Maximum size of the workpiece 760x400x500mm,

Maximum weight of the billet is 1300 kg,

Positioning accuracy ± 0,005 mm,

Positioning repeatability ± 0,0025 mm,

Indexed rotary axis

CNC EDM (electrical discharge) wire-cutting machine Mitsubishi MV1200S

Main parameters and features:

- Maximum size of the billet 810x700x215mm
- Maximum weight of the billet is 500 kg
- The achievable accuracy on the part is 0.0025 ± mm
- The best roughness is Ra, 0.25 µm

Types of processed materials:

- Processing of any metals, alloys, electrically conductive nonmetallic materials, incl. high hardness.
- Ferrous and non-ferrous metals and alloys, stainless and special steels, hardened steels, graphite, hard alloy (tungsten carbide), cubic boron nitride (CBN), polycrystalline diamond.

EDM start hole drilling machine Advanced Machinery EDM AD24

Main parameters and features:

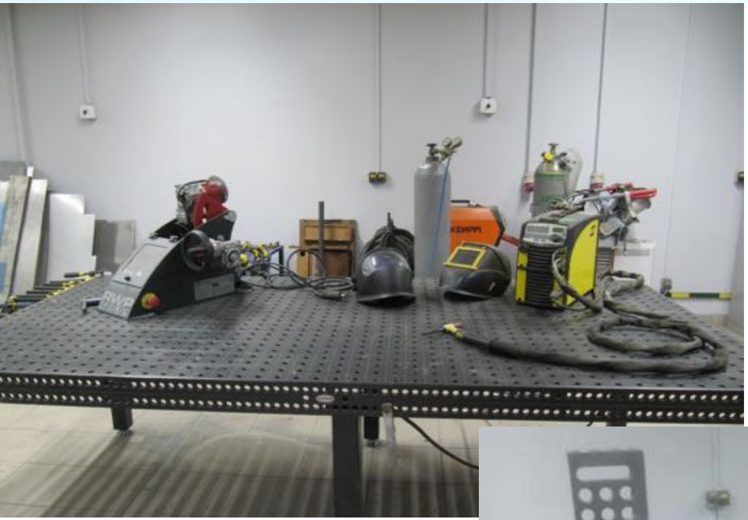
- Maximum size of the billet 810x510x240mm
- Maximum weight of the billet is 300 kg
- The achievable accuracy on the part is 0.0025 ± mm
- The best roughness is Ra, 0.25 µm
- Diameters of used electrodes 0,3-3 mm

Types of processed materials:

- Processing of any conductive materials

Examples of manufactured parts

Welding equipment and works
Welding workshop



Weldable materials:

- Stainless steel
- Copper
- Aluminium allous
- Titanium

Technologies:

- TIG welding
- MIG welding
- MMA welding
- Rotary welding with CNC welding table (rotator)

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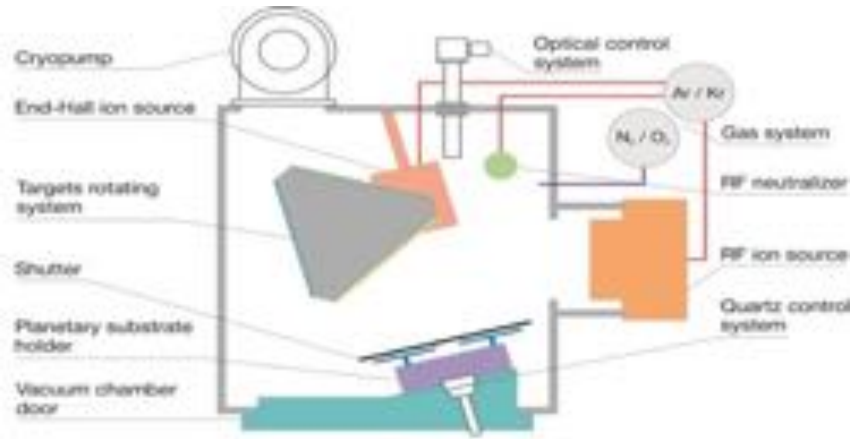
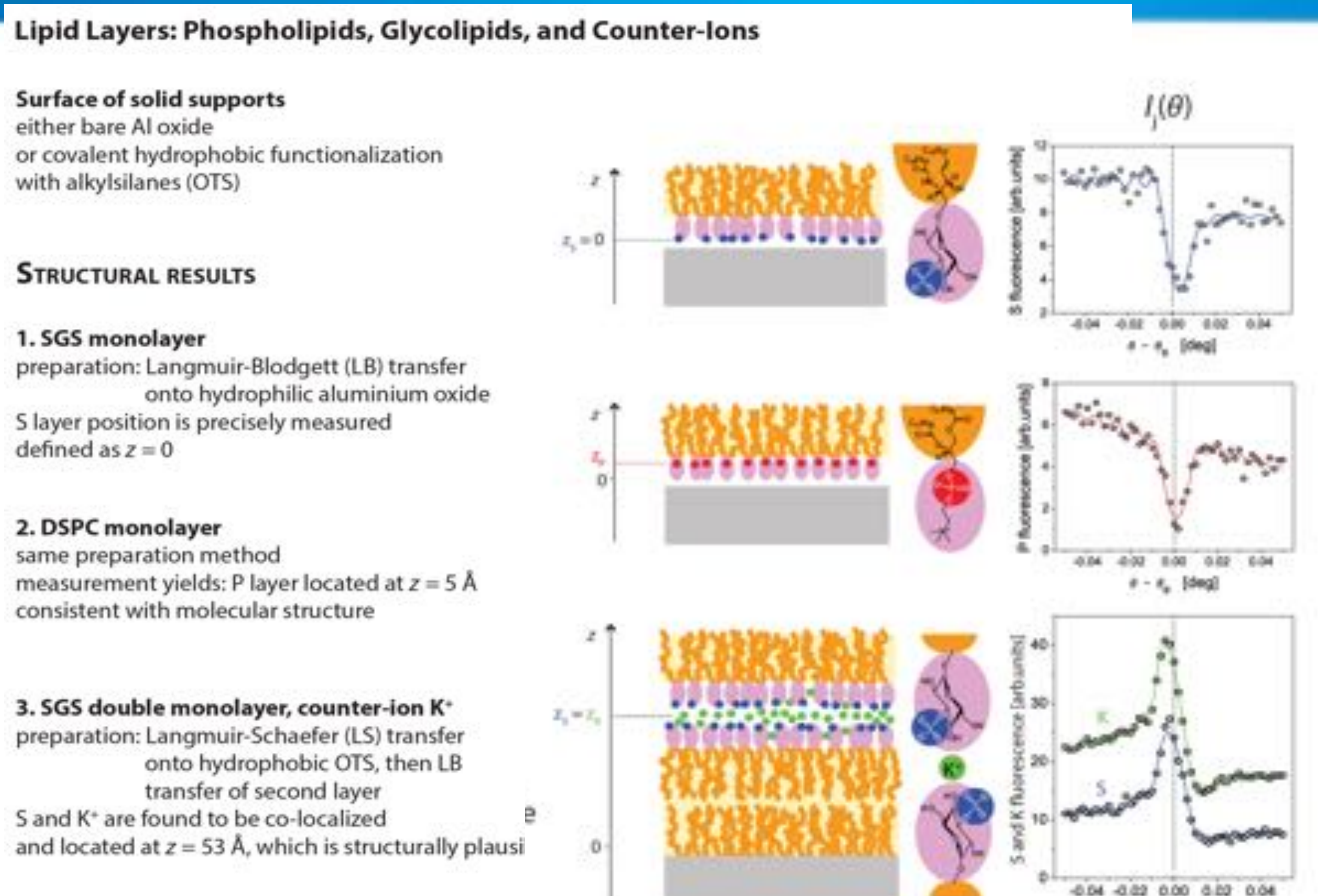
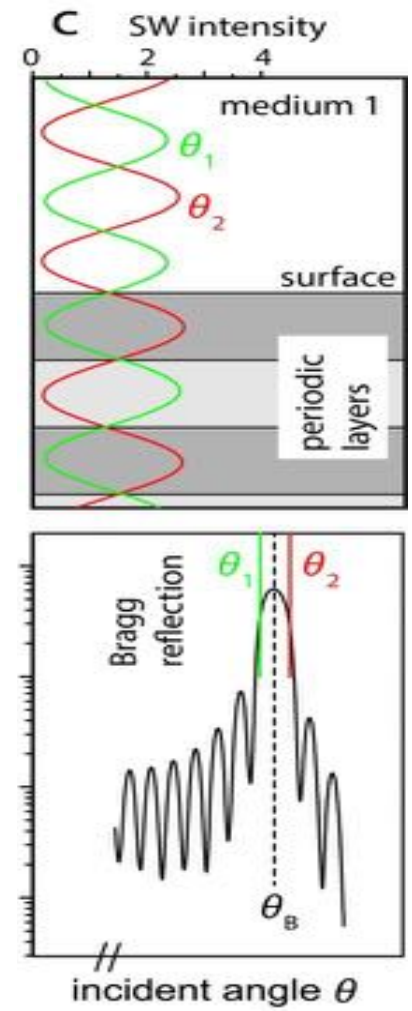
Ion Beam Deposition Complex

- Vacuum technology (operating at 10^{-2} Pa)
- RF gridded Kaufman Ion Source;
- End-Hall griddles assisting source;
- 3-targets rotating holder;
- planetary rotating substrate holders;
- Optical and quartz thickness control;
- Ar and Kr as source gases;
- O_2 and N_2 operations.



Al/Ni (2.5 nm, 25 periods) for MPI-CI



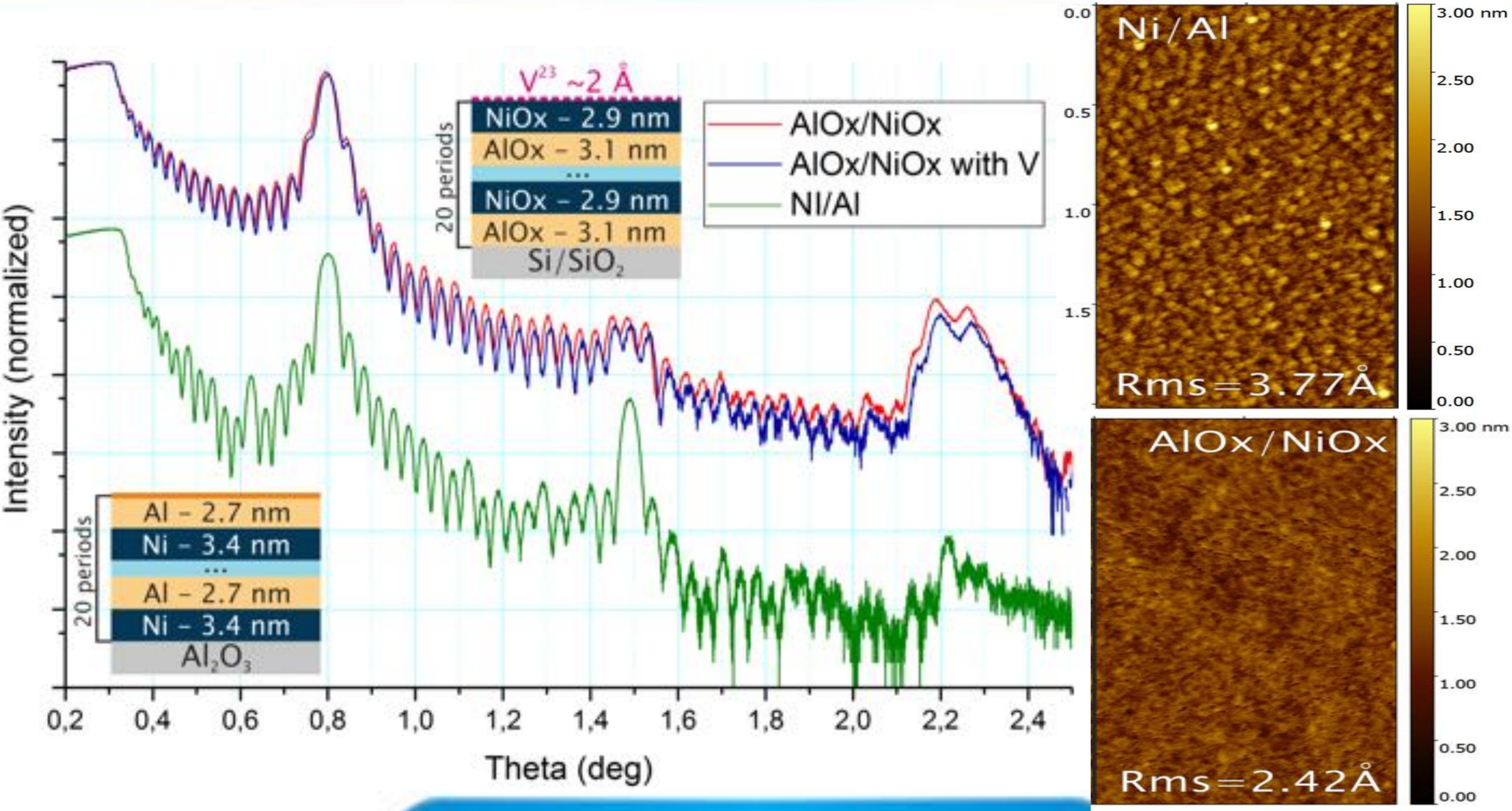


“Structural Characterization of Soft Interfaces by Standing-Wave Fluorescence with X-Rays and Neutrons”

By E.Schneck and B.Demé.

J. of Colloid and Interface Sci., 2015

Al/Ni (2.5 nm, 20 periods)



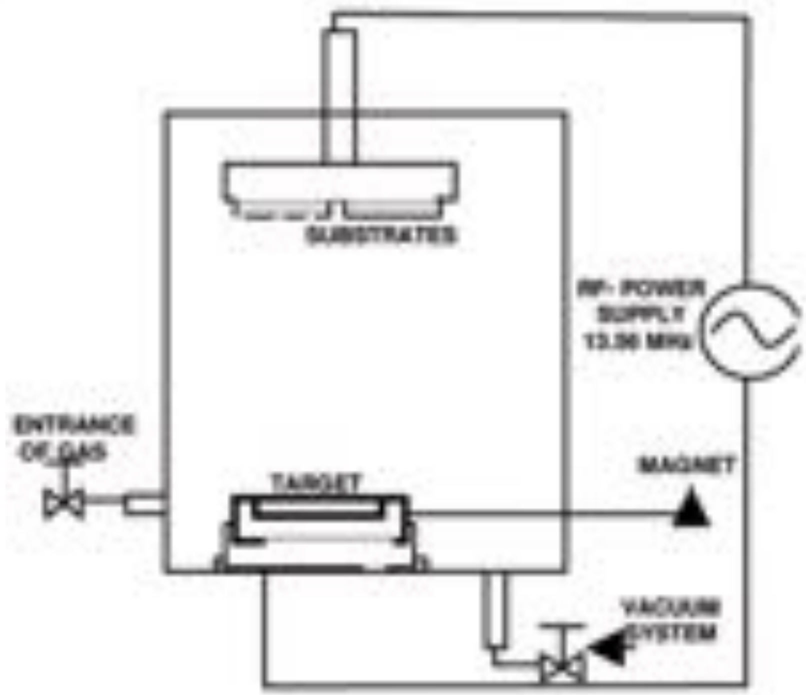
Large Area RF Magnetron Sputtering

Main characteristics

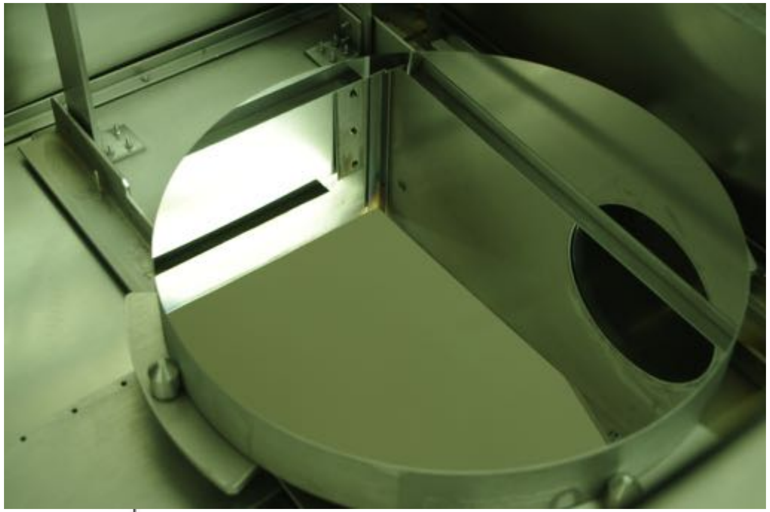
- RF Magnetron Sputtering
- 10 kW
- 1x1 m deposition area
- Vacuum system 1000 l/s
- Al, Ag, Cu, Ti coatings
- Oxides (SiO2, AlOx etc.)
- Nitrides (TiN. AlN)

RF Magnetron

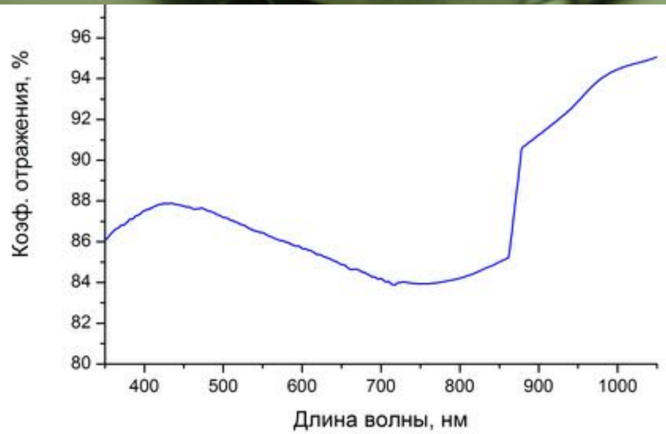
RF Matching network (antenna)



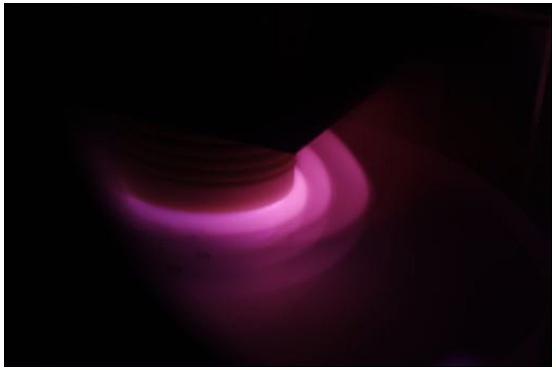
50 cm dia optical Al/SiO_2 mirrors for space applications by RF magnetron



Optical quartz substrate
⃠ 500mm, d = 65mm., 30 kg
High quality polishng
N<1
RF Magnetron coating Al(50nm)/SiO2, K>85%



Plasma cleaning



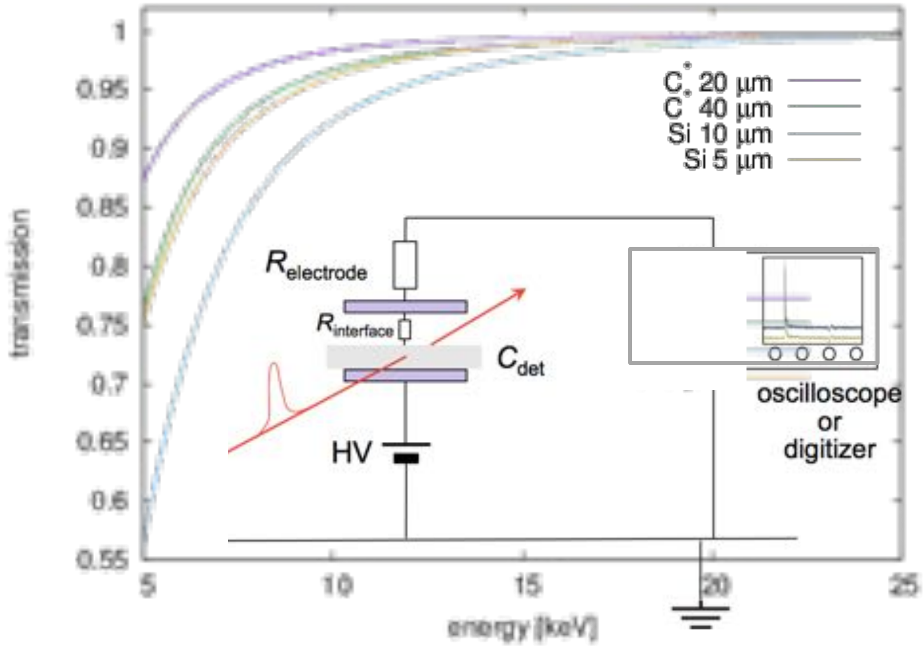
Thin diamond detectors for XFEL

+ lowest-Z detector:
- most ablation hard
- low absorption

+ high heat conductivity
+ high melting point
+ insensitive to visible light
+ fast charge drift velocities
+ compact

- small: scCVD about 4.5 x 4.5 mm^2
- still rather thick: 25-40μm thickness
- expensive
- require bias voltage

"ionisation chambers - like" detectors:



* Materials Imaging and Dynamics end-station task by Andres Madsen

Thin diamond detectors for XFEL

MID station XFEL task: Be coated detectors

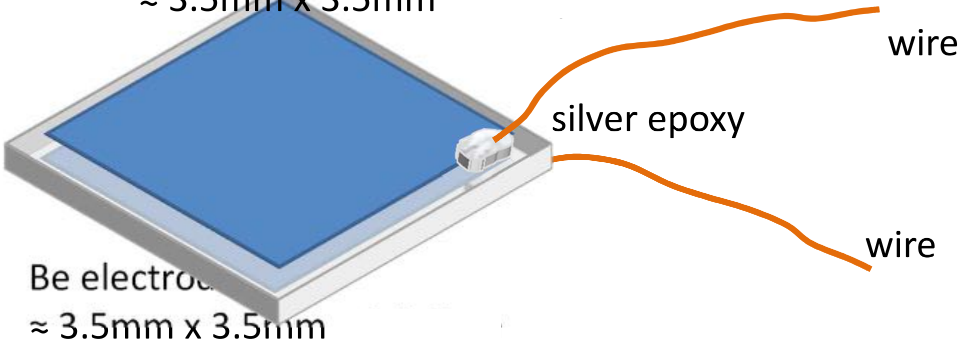
- The thickness should be 0.5-2 microns each side, see attached figure.
- There should not be any intermediate layer between diamond and Be.
- There should not be Beryllium on the edges as we can not allow an electric contact of the two electrodes via Beryllium on the edges

+lowest-Z electrode ma

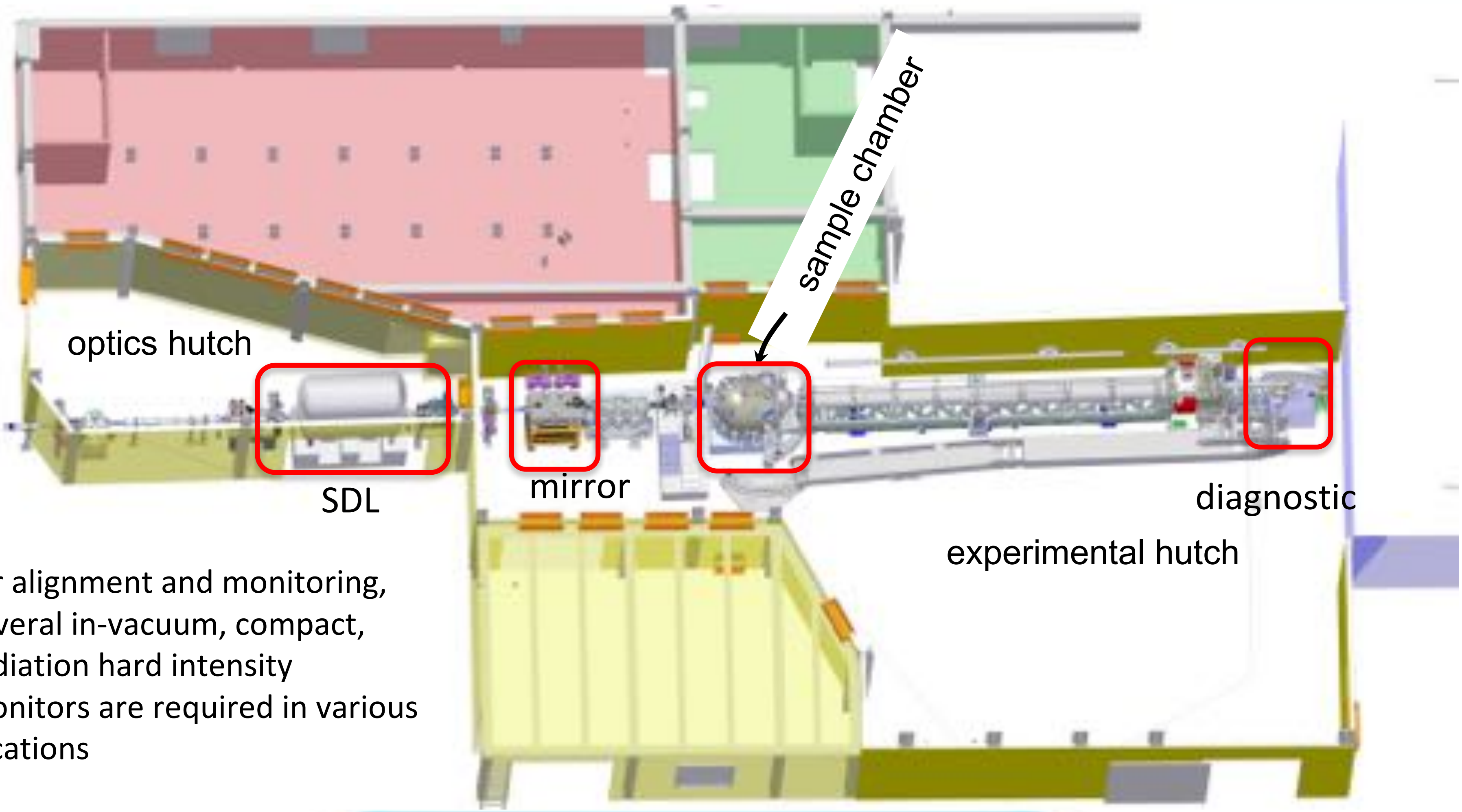
- most ablation hard

+ metallic

- small resistivity,
- thus very fast time c

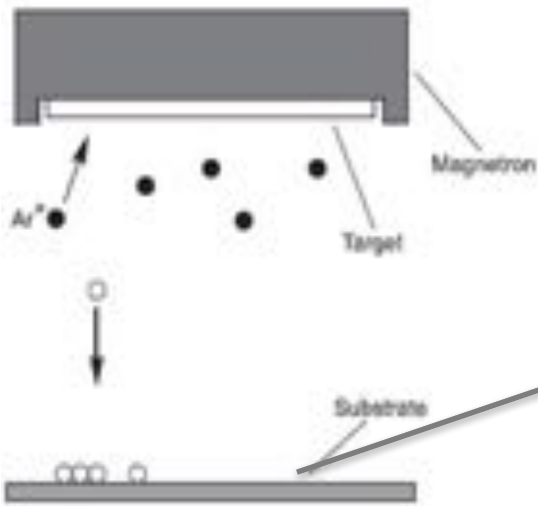


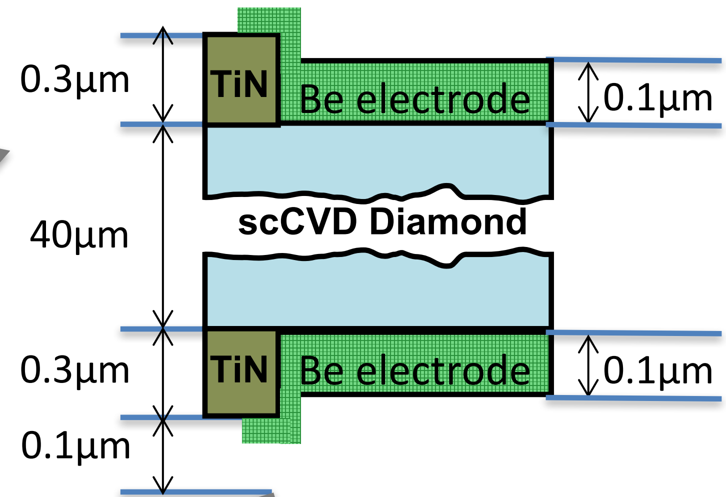
Optics and experimental hutches of the MID end-station

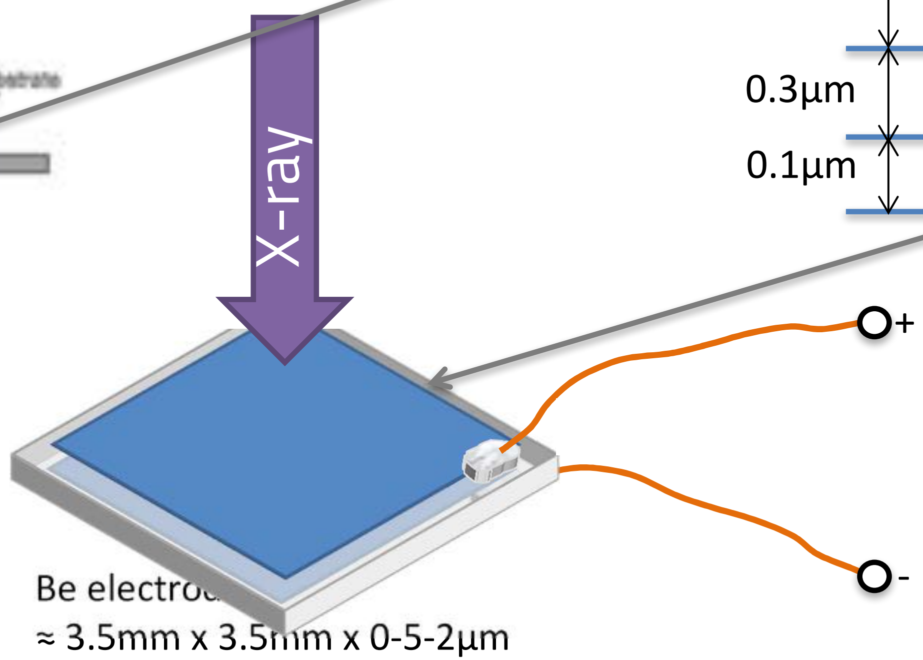


for alignment and monitoring, several in-vacuum, compact, radiation hard intensity monitors are required in various locations

Be/scCVD-Diamond/Be device



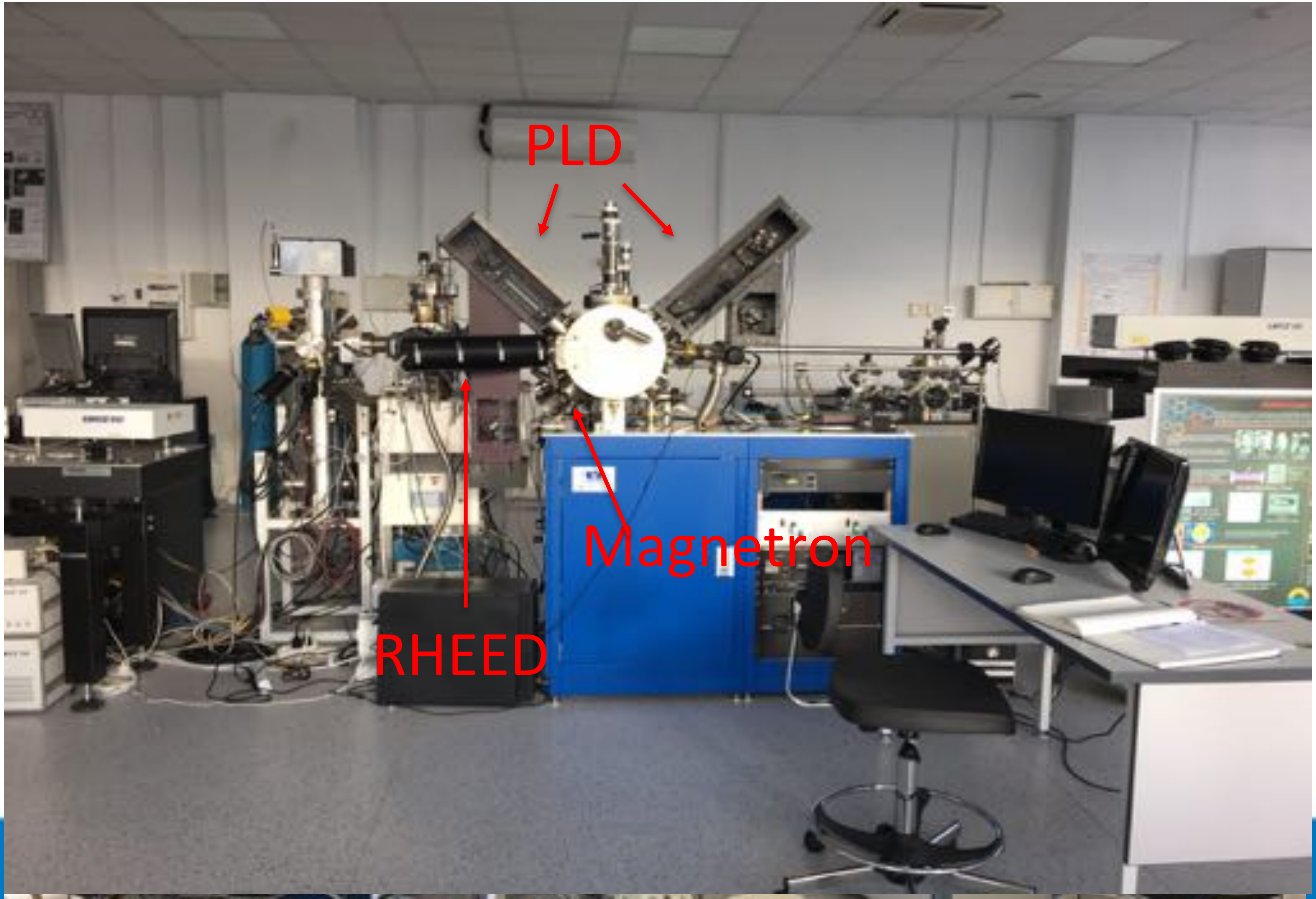




scCVD diamond plate,
4mm x 4mm x 40µm

J. Synchrotron Rad. (2018). **25** https://doi.org/10.1107/S1600577517015016
T. Roth et al.

Единый комплекс вакуумных ростовых и исследовательских установок



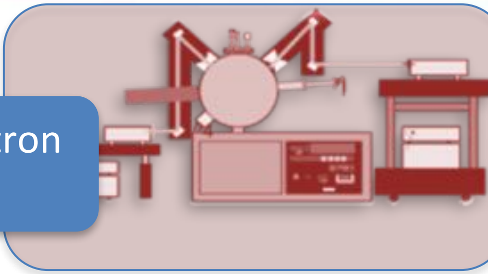
Home lab

Thin film formation & investigation *in-situ* complex

Pulsed laser deposition

Low energy electron dif-fraction

Auger electron analysis



Atomic Layer Deposition

Magnetron sputtering

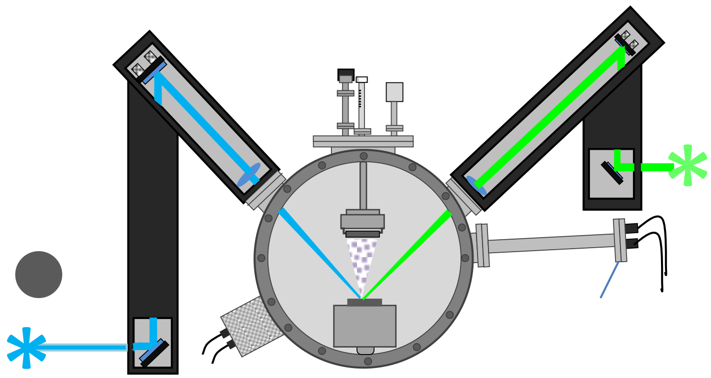
Sample pre-paration

UHV Atomic forc e microscope

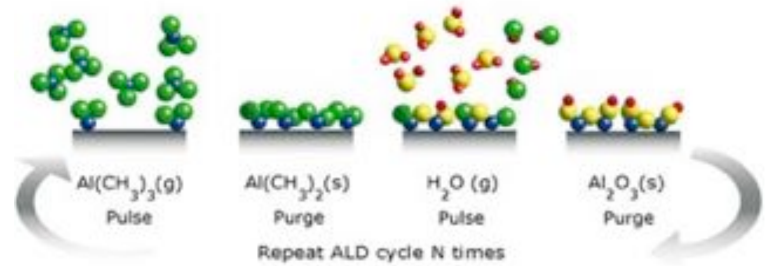
Time of Flight mass-spectroscopy

Refractive high energy electrons diff

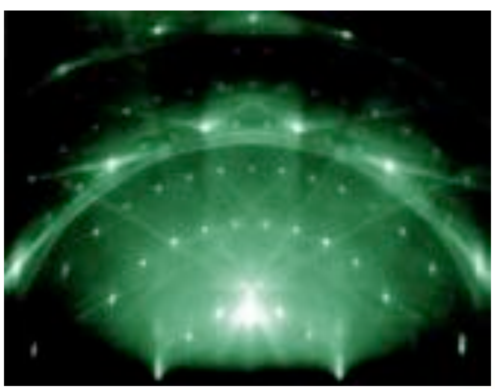
Thin film formation & investigation *in-situ* complex



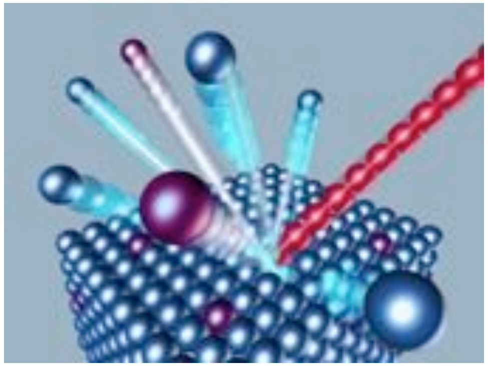
2 Nd:YAG lasers co-deposition system



Atomic layer depostion in situ



Refractive high energy electrons diffraction



Time of Flight secondary ion mass-spectroscopy

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Thin film formation & investigation *in-situ* complex

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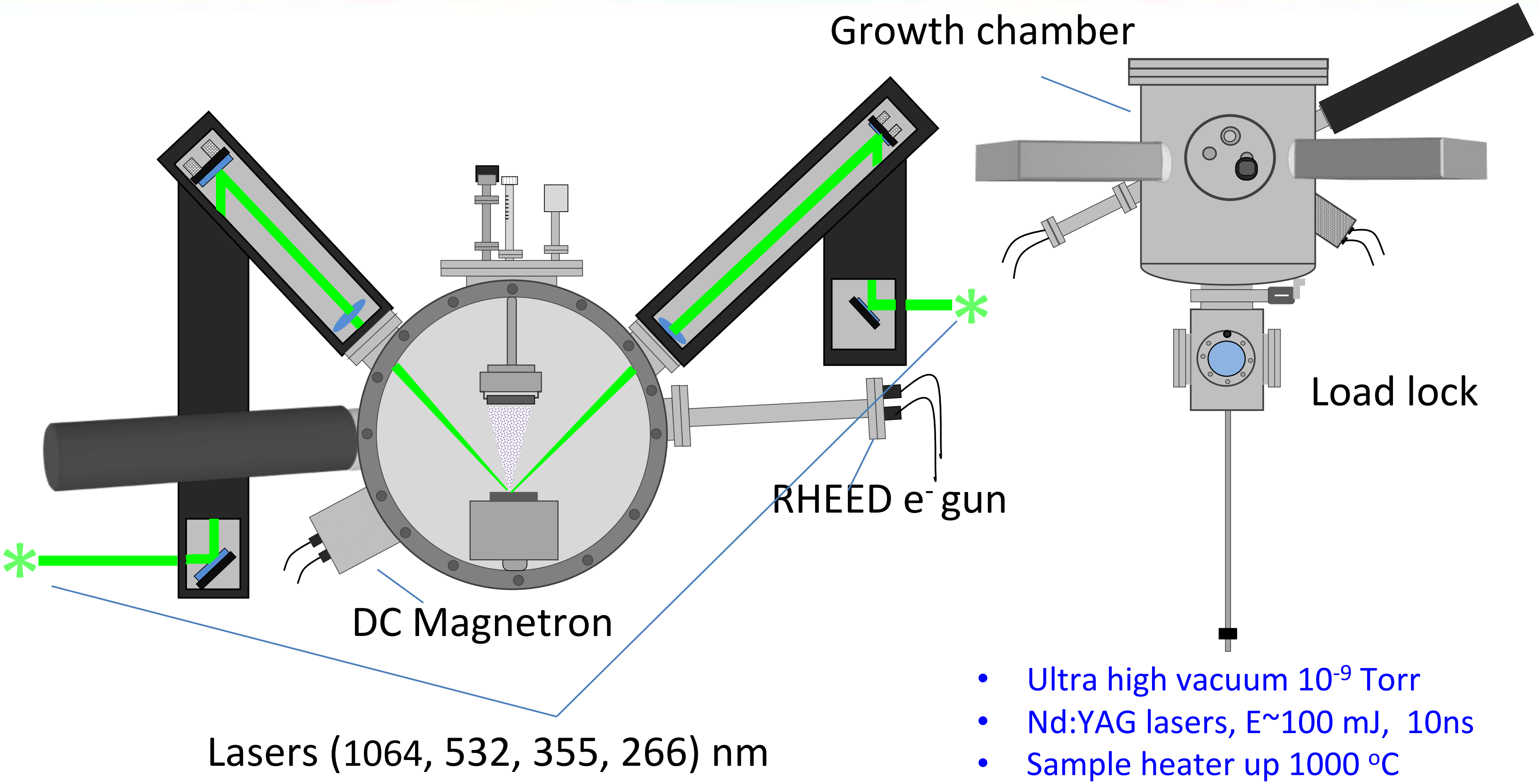
Experimental PLD Setup



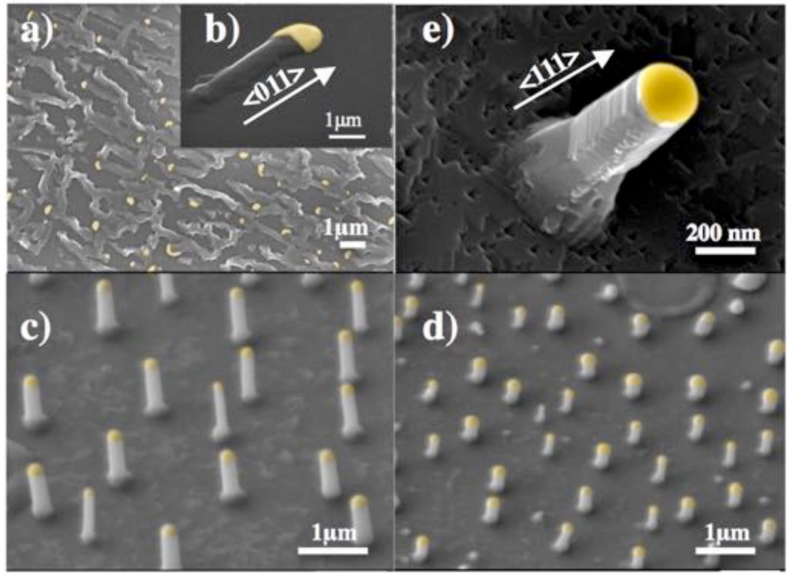
- Ultra high vacuum 10^{-9} Torr
- Nd:YAG lasers, E~100 mJ, 10ns
- Sample heater up 1000 °C
- Magnetic Field annealing

Si:Au nanowhiskers for nanoelectronic devices

GROWTH METHOD

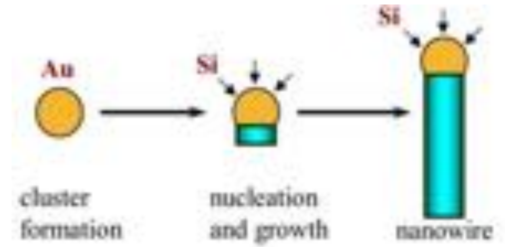
Pulsed Laser Deposition (PLD)

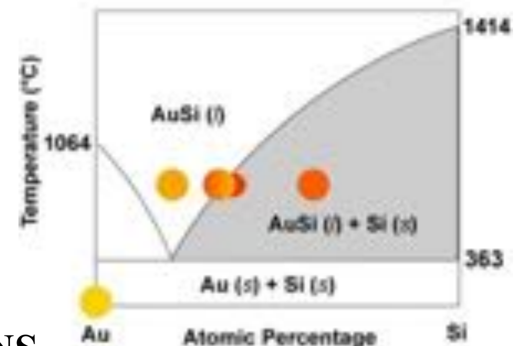
- High particles energy in the plasma plume (up to 50keV);
- Precise thickness control (~0.1ML/pulse)
- realization of experiment in the different gas atmosphere (O_2: Ar, N_2);
- Laser harmonics: 1064nm; 532nm; 355nm; 266nm;
- Possibility growth heterostructures in one vacuum cycle;



GROWTH MECHANISM

vapor–liquid–solid method (VLS)



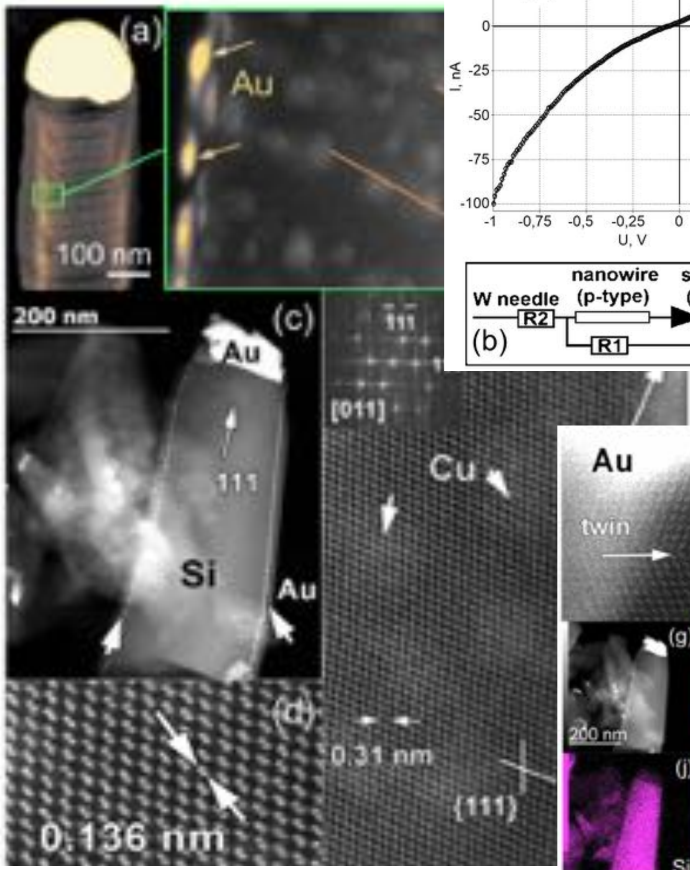


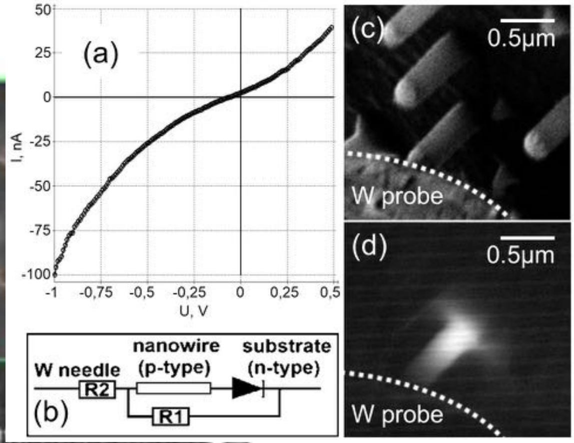
CONCLUSIONS

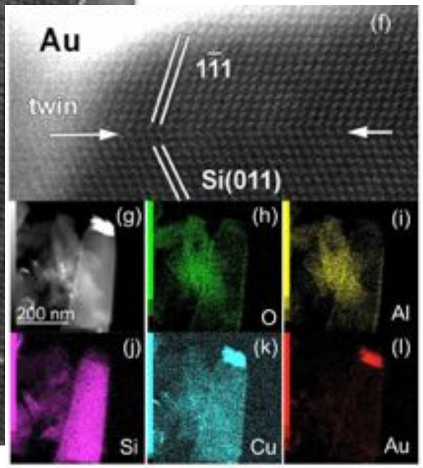
- Possibility of gold clusters formation by PLD. Higher substrate temperature – larger size of the separate cluster.
- Au clusters crystallized in (111) orientation;
- Si:Au NW is possible to growth by PLD on Si(111) substrate in a special conditions (vac. pressure, laser energy, …).

Copper-stabilized Si:Au nanowhiskers for advanced nanoelectronics application

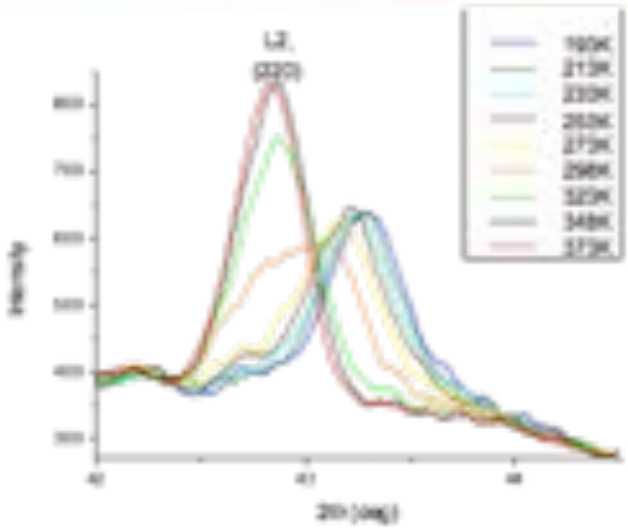
- For the first time, we demonstrate that this method could be employed to control the size and shape of silicon NWs by using a two-component catalyst material (Cu:Au~1:60).
- During the NW growth, copper is distributed on the outer surface of the nanowhisker, while gold sticks as a droplet to its top.
- The measurements of the electrical transport properties revealed that in contact with the substrate, individual NWs demonstrate typical I-V diode characteristics.

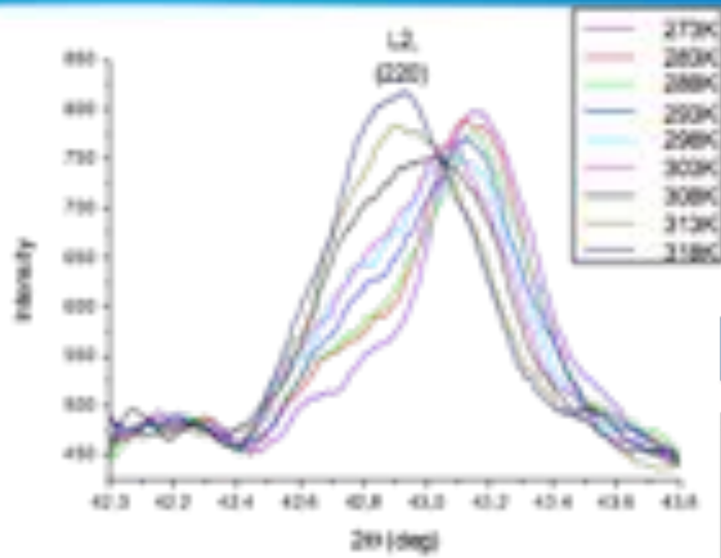


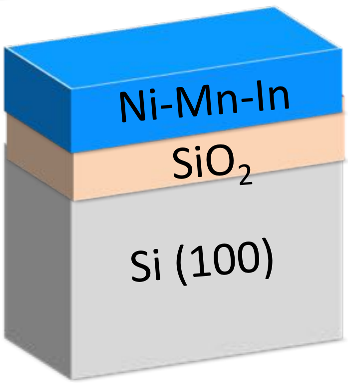


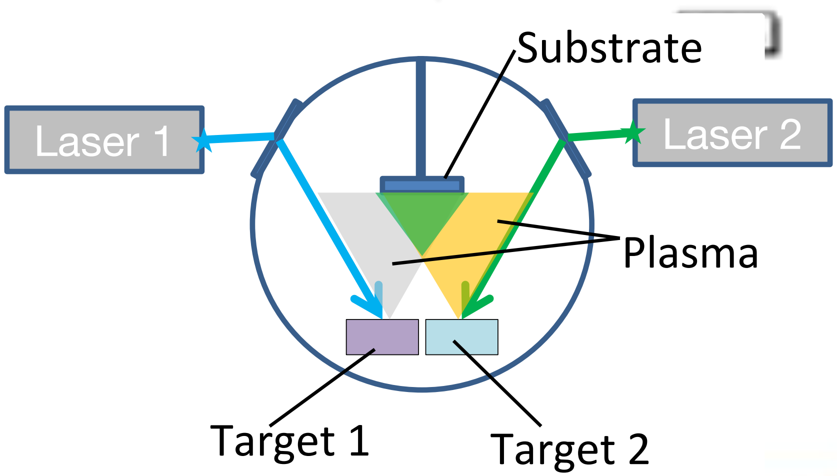


Thin $Ni_{51}Mn_{33}In_{16}$ film XRD investigation







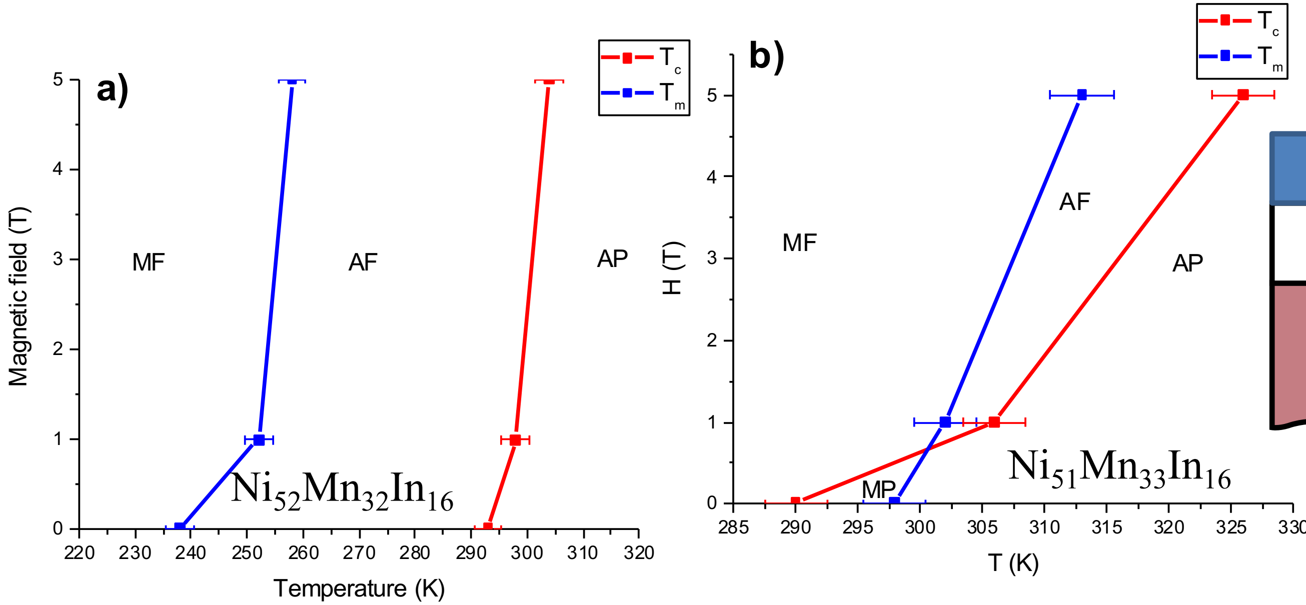


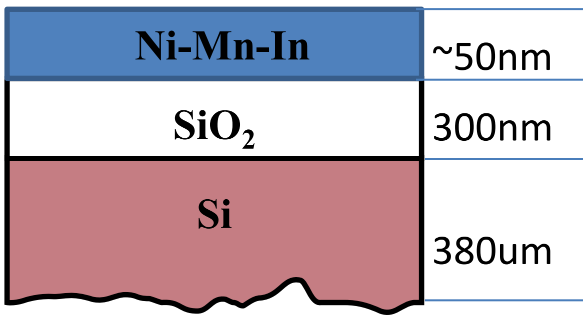
- Near-room martensitic transition.
- Narrow temperature interval of the martensitic transition (~30K).
- Transition temperature ~310K.

by Alexey Grunin

Magnetostructural transformations in Ni-Mn-In thin films

Properties of martensitic transition are investigated by X-Ray diffraction at different temperatures, magnetometery and HAXPES





Phase diagrams for samples $Ni_{52}Mn_{32}In_{16}$ (a) u $Ni_{51}Mn_{33}In_{16}$ (b)
M – martensitic phase, A – austenite. F – ferromagnetic, P - paramagnetic

*Alexei Grunin thesis defense 25.10.17

Functional Nanomaterials

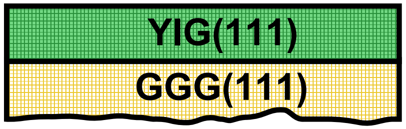
YIG(111)/GGG(111) Structures by PLD

Thin yttrium iron garnet films grown by pulsed laser deposition: Crystal structure, static, and dynamic magnetic properties

N. S. Sokolov,[1,a)] V. V. Fedorov,[1] A. M. Korovin,[1] S. M. Suturin,[1] D. A. Baranov,[1] S. V. Gastev,[1] B. B. Krichevtsov,[1] K. Yu. Maksimova,[2] A. I. Grunin,[2] V. E. Bursian,[1] L. V. Lutsev,[1] and M. Tabuchi[3]
[1]*Ioffe Physical-Technical Institute of Russian Academy of Sciences, St. Petersburg 194021, Russia*
[2]*Immanuel Kant Baltic Federal University, Kaliningrad 236041, Russia*
[3]*Synchrotron Radiation Research Center, Nagoya University, Nagoya 464-8603, Japan*

(Received 26 October 2015; accepted 27 December 2015; published online 12 January 2016)

Pulsed laser deposition has been used to grow thin (10–84 nm) epitaxial layers of Yttrium Iron Garnet $Y_3Fe_5O_{12}$ (YIG) on (111)–oriented Gadolinium Gallium Garnet substrates at different growth conditions. Atomic force microscopy showed flat surface morphology both on micrometer and nanometer scales. X-ray diffraction measurements revealed that the films are coherent with the substrate in the interface plane. The interplane distance in the [111] direction was found to be by 1.2% larger than expected for YIG stoichiometric pseudomorphic film indicating presence of rhombohedral distortion in this direction. Polar Kerr effect and ferromagnetic resonance measurements showed existence of additional magnetic anisotropy, which adds to the demagnetizing field to keep magnetization vector in the film plane. The origin of the magnetic anisotropy is related to the strain in YIG films observed by XRD. Magneto-optical Kerr effect measurements revealed important role of magnetization rotation during magnetization reversal. An unusual fine structure of microwave magnetic resonance spectra has been observed in the film grown at reduced (0.5 mTorr) oxygen pressure. Surface spin wave propagation has been demonstrated in the in-plane magnetized films.
 [http://dx.doi.org/10.1063/1.4939678]



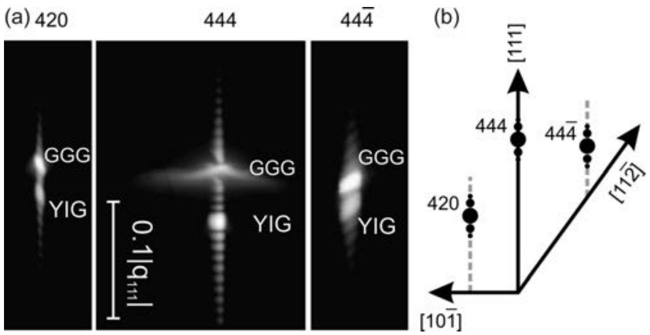
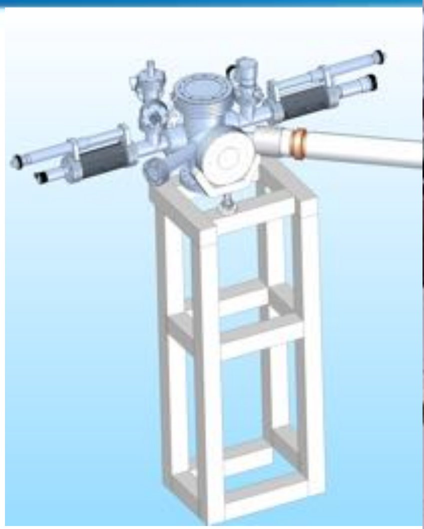
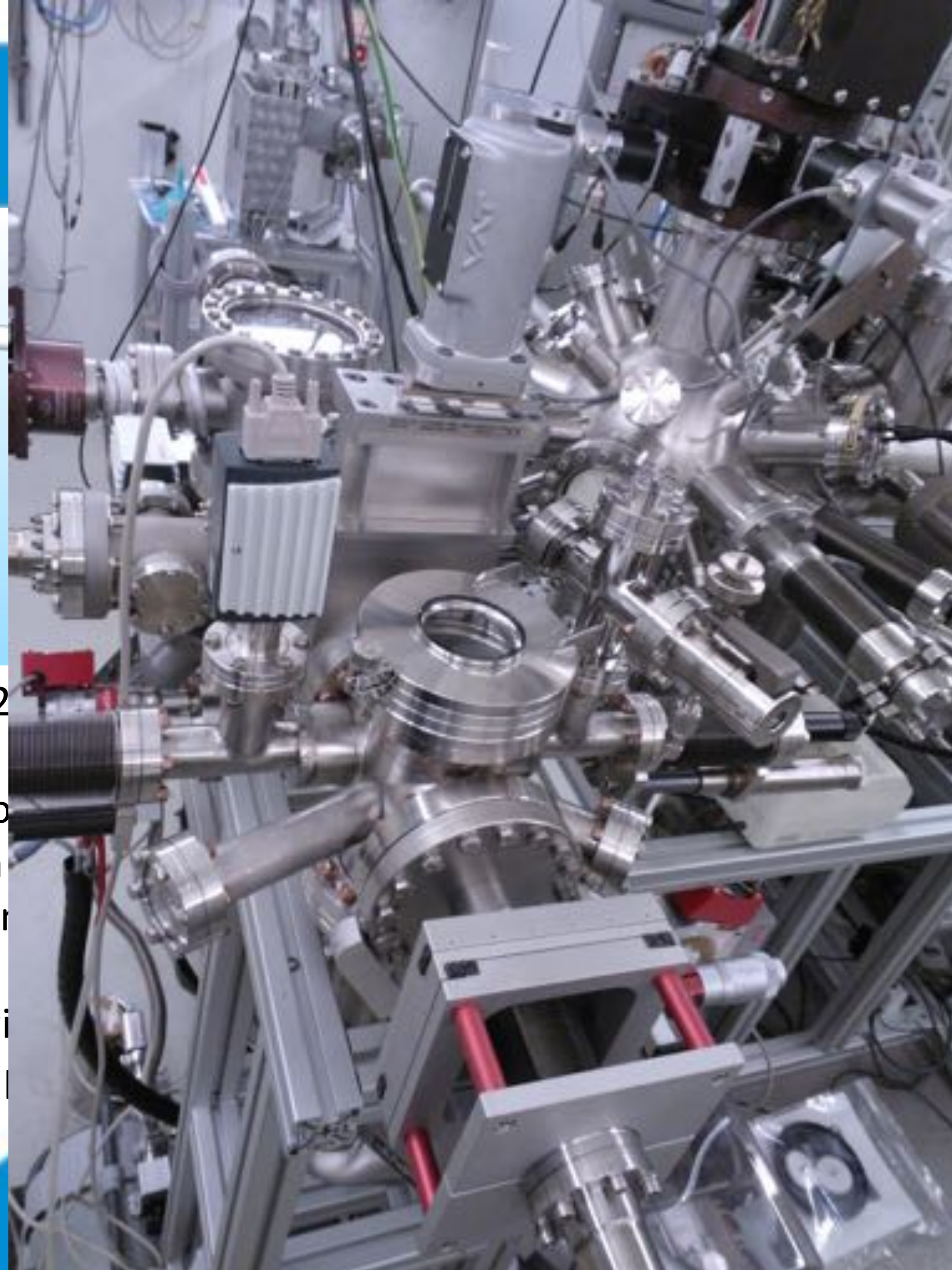


FIG. 2. 3D reciprocal space maps in the vicinity of GGG (420), (444), and (44-4) reflections of the sample with 84 nm YIG film (a) and schematic drawing of the corresponding crystal truncation rods (b).

The origin of the magnetic anisotropy is related to the strain in $\mathbf{Y_3Fe_5O_{12}}$ films observed by XRD

Functional Nanomaterials



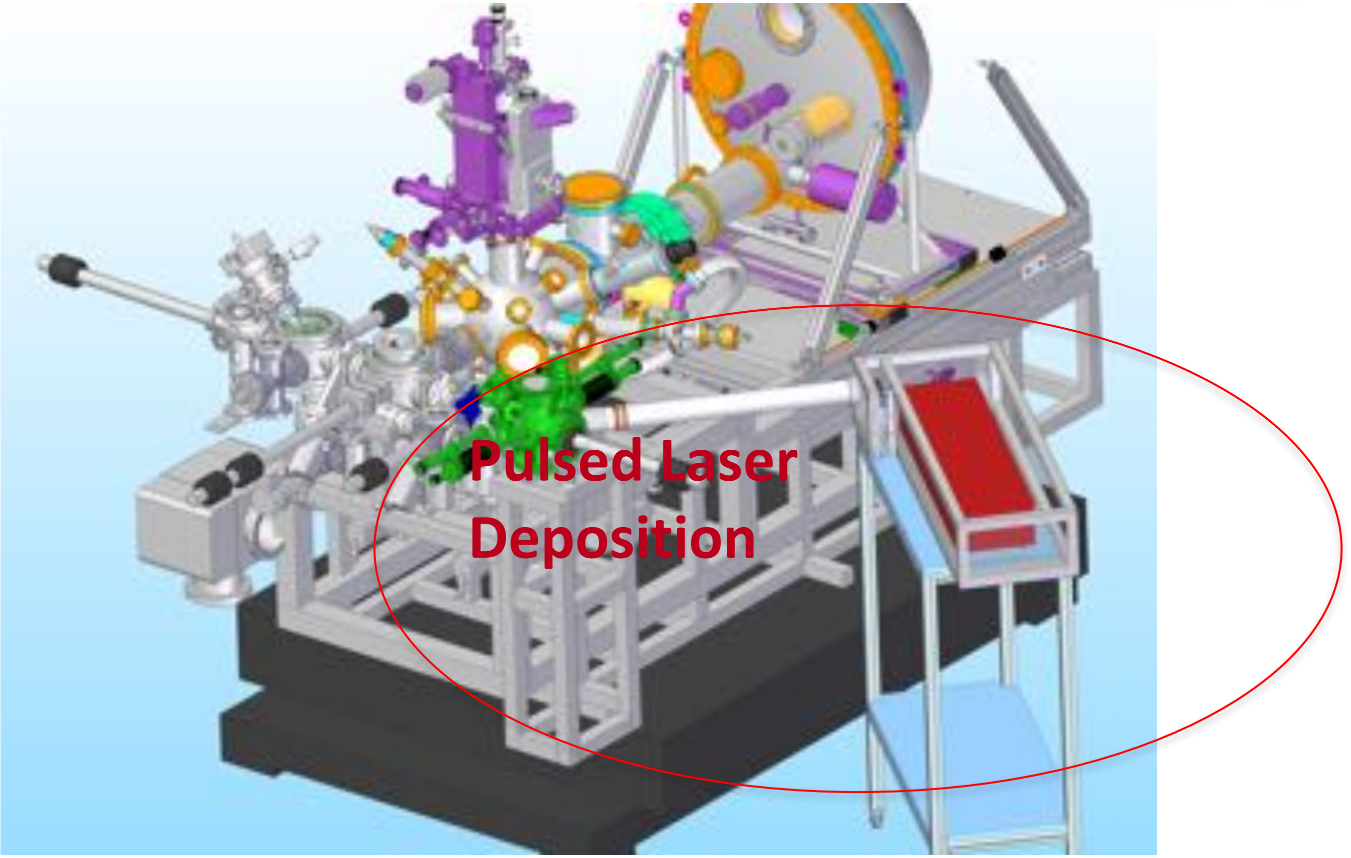


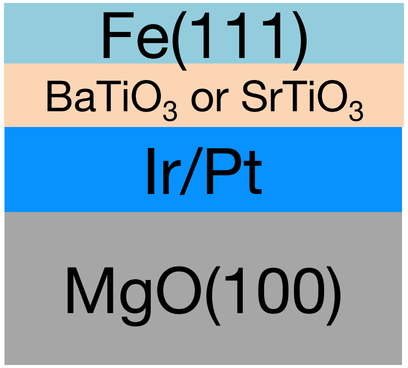
- Laser: 266nm, 532
  + focusing lens;
- Electrical feedthro
- Variable precision
- Linear and rotary
  and target;
- Vacuum gauge (wi
- Agilent TPS turbo

ifferent

ith lamp

PLD exp @ P09 PETRA III. Постановка эксперимента



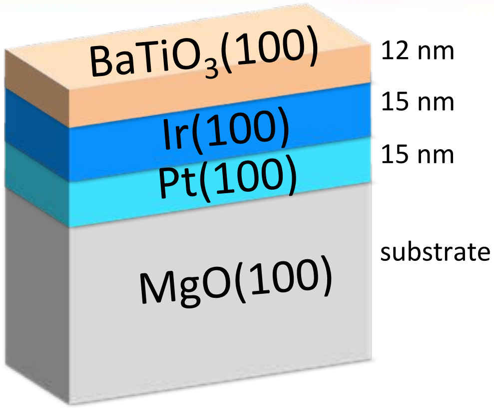


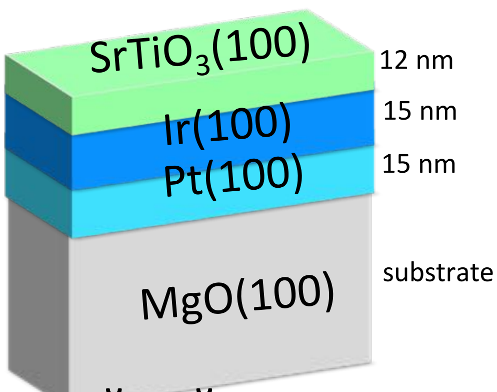
- Импульсное лазерное осаждение: Высокие энергии(50 eV до 500 eV) ➔ большая длина диффузии на поверхности ➔ монокристаллы

by Ksenia Maksimova

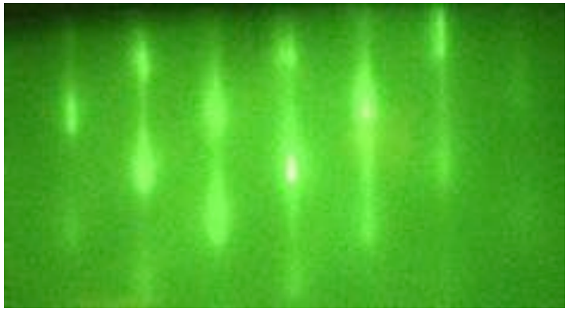
PLD exp @ P09 PETRA III. Структурные свойства

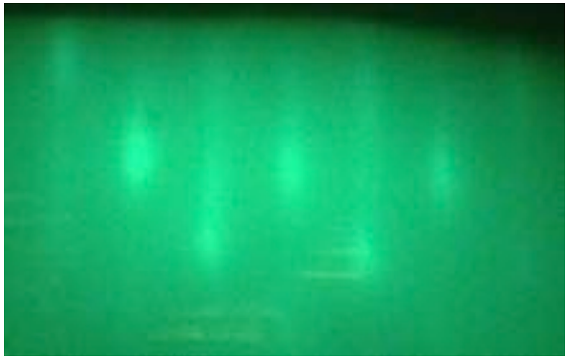
BTO and STO tetragonal



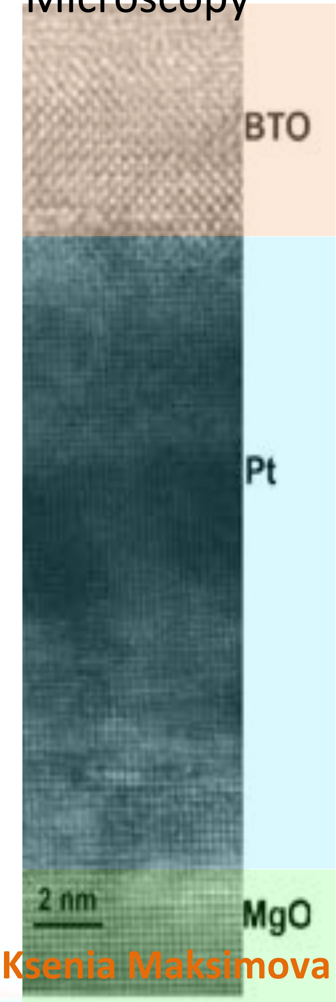


Reflection High Energy Electron Diffraction





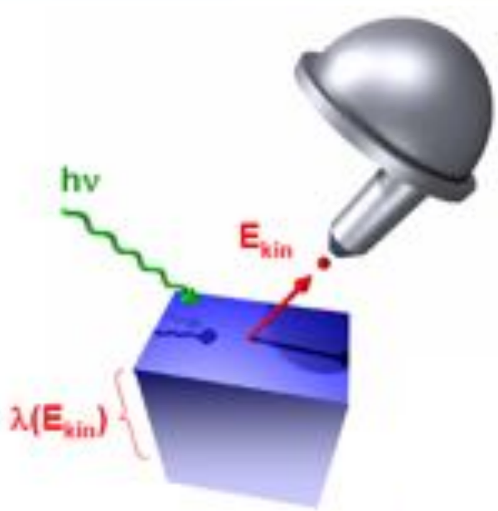
Transmission Electron Microscopy

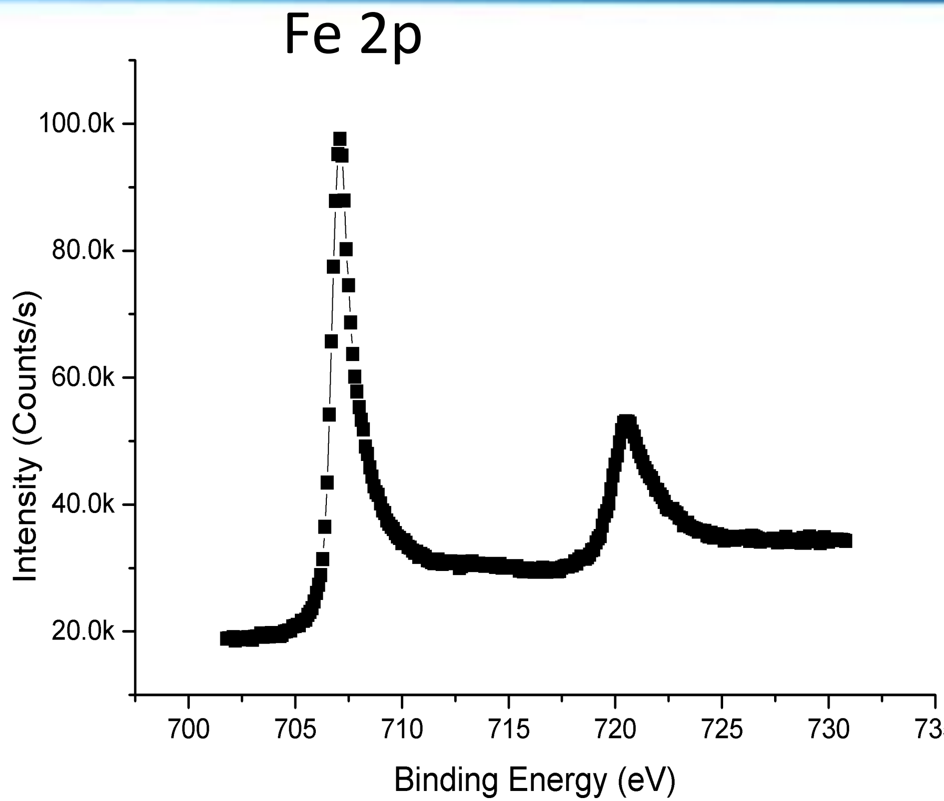


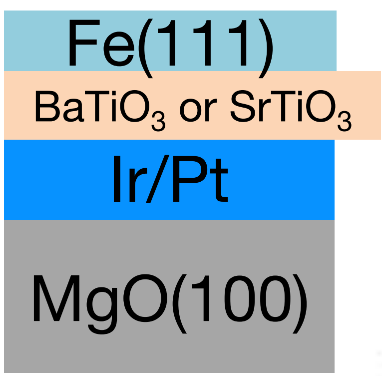
by Ksenia Maksimova

- Послойный рост с гладкими границами раздела;
- Несоответствие решеток $BaTiO_3$/Ir= 2.1%; Fe/$BaTiO_3$= 3.3%;
- Несоответствие решеток $SrTiO_3$/Ir= 1.7%; Fe/$SrTiO_3$= 3.7%;

PLD exp @ P09 PETRA III. Фотоэлектронная спектроскопия





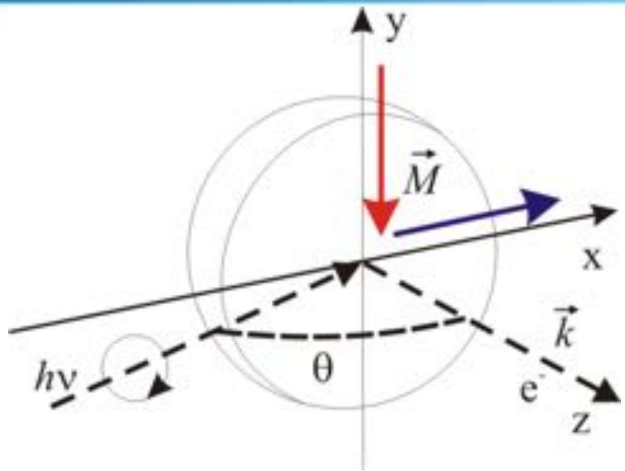


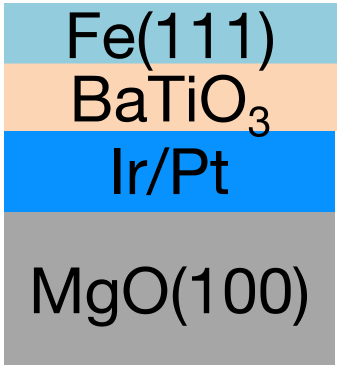
- Границы раздела Fe/BTO, Fe/STO не содержат оксид железа

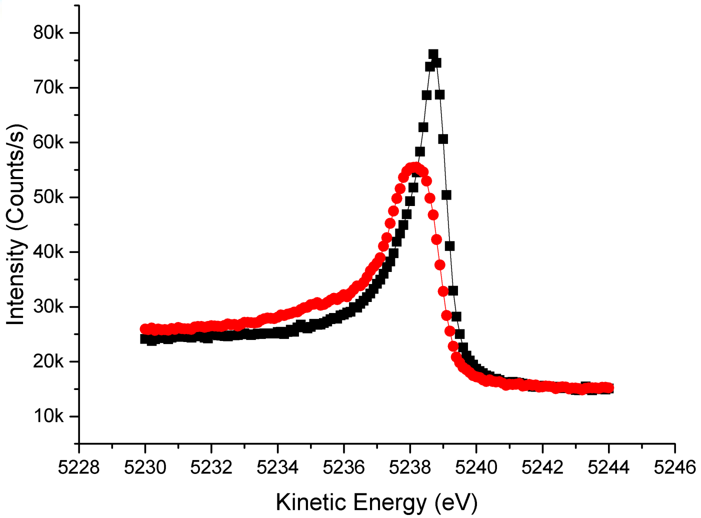
by Ksenia Maksimova

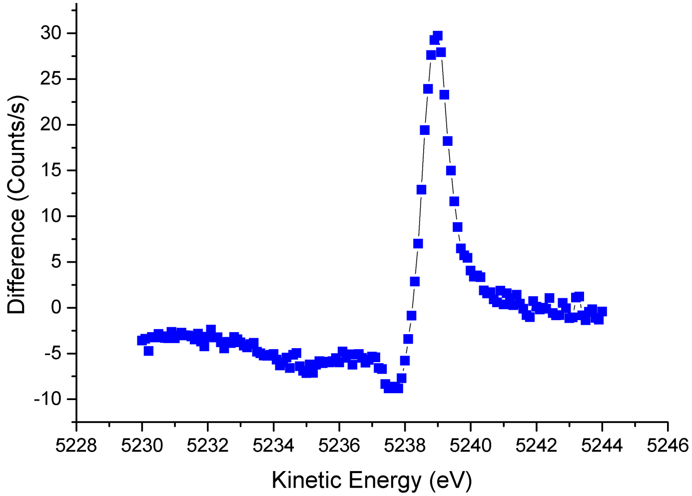
Functional Nanomaterials

PLD exp @ P09 PETRA III. Магнитный круговой дихроизм









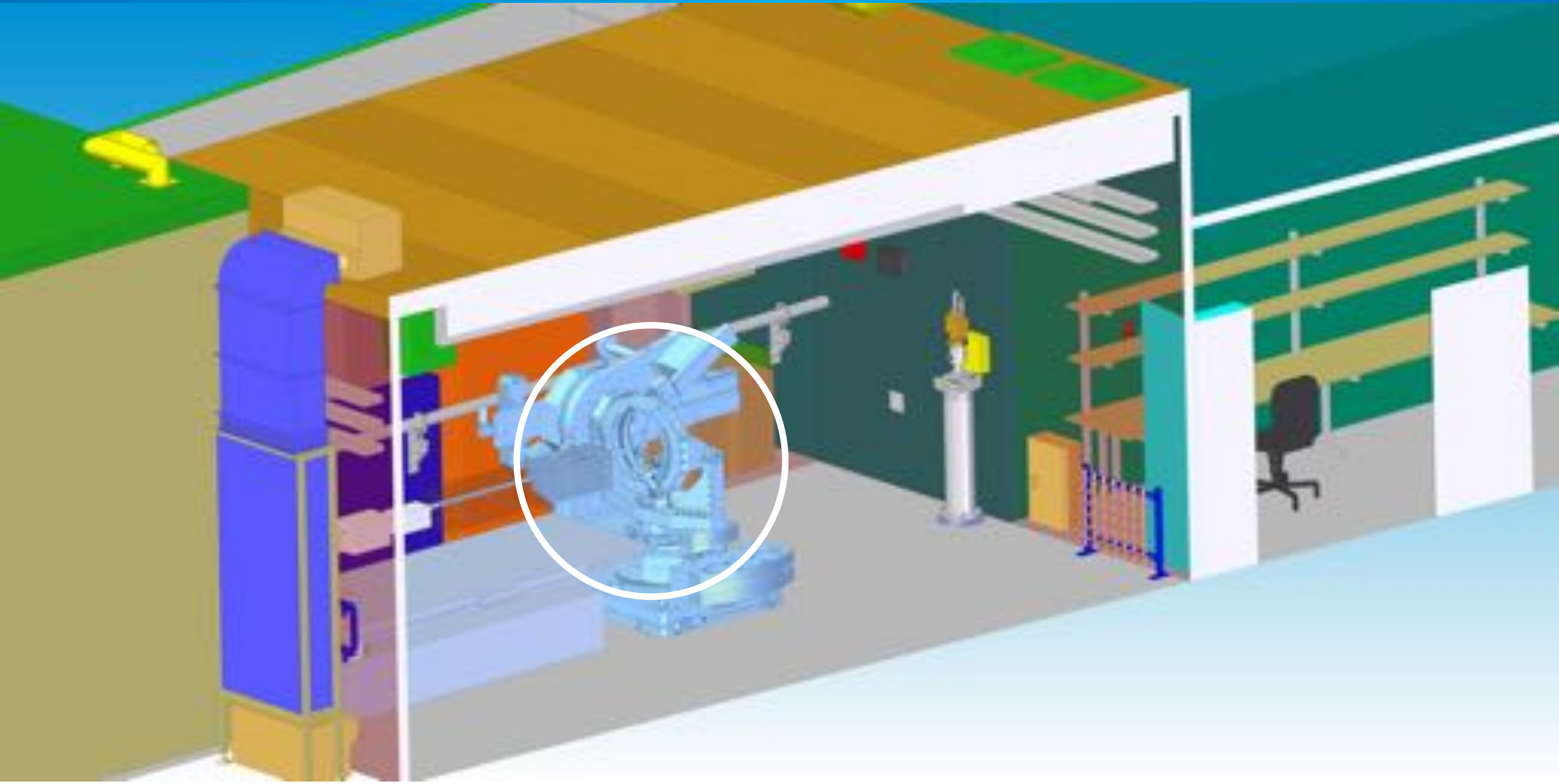
Интенсивность и форма спектров фотоэмиссии зависит от относительной ориентации и / или направления:

- Вектора намагничивания образца
- Поляризации рентгеновского излучения
- Эмиссии электронов

by Ksenia Maksimova

Functional Nanomaterials

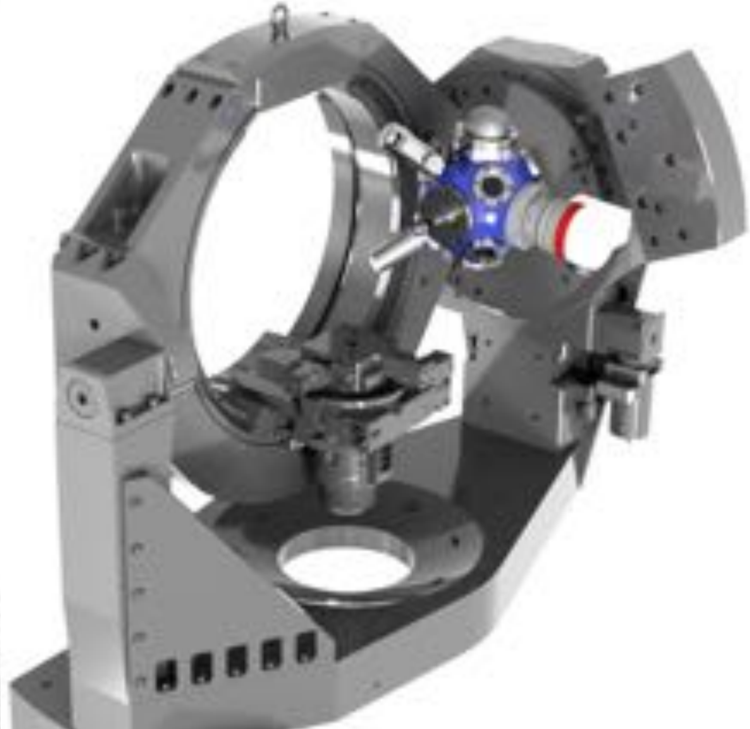
DESY, PETRA III, P23 (Russian-German)



- Thin film deposition system concepts for *in situ* investigations

Мобильные ростовые установки: DESY, P23 Russian-German Beamline: thin film growth concept

Российско-Немецкий канал P23

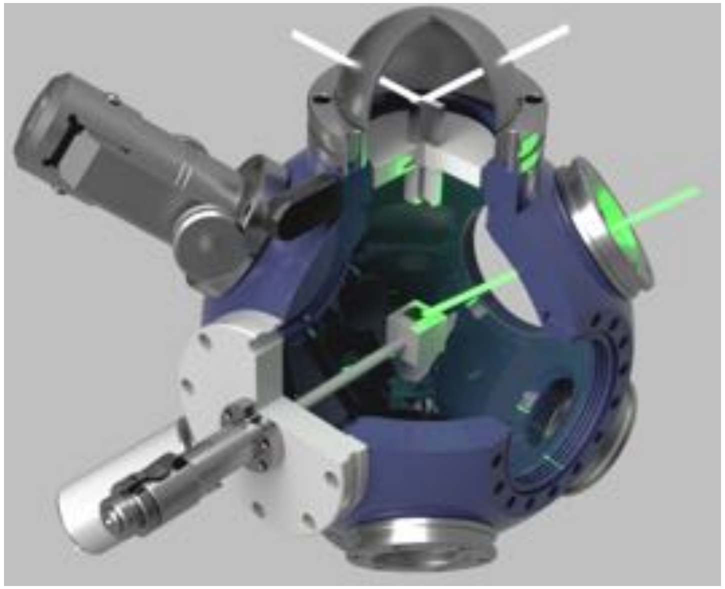


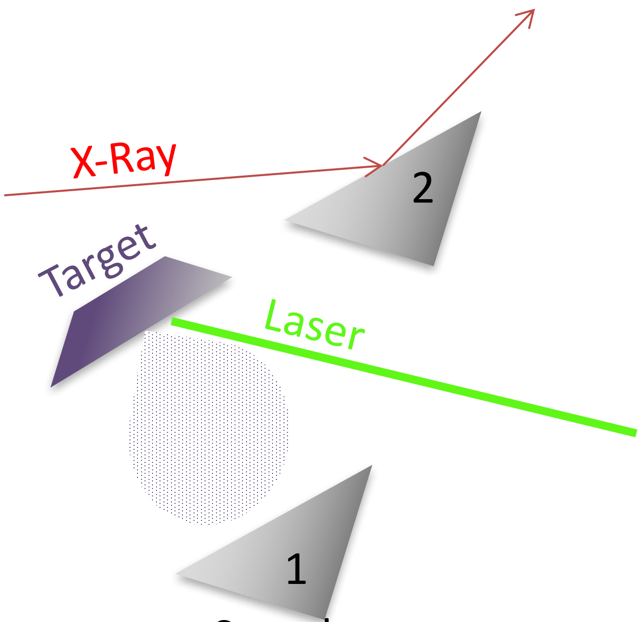
Гониометр EH1, размещение легкой ростовой камеры

by Ksenia Maksimova

Functional Nanomaterials

Мобильные ростовые установки: DESY, P23 Russian-German Beamline: thin film growth concept





Main features:

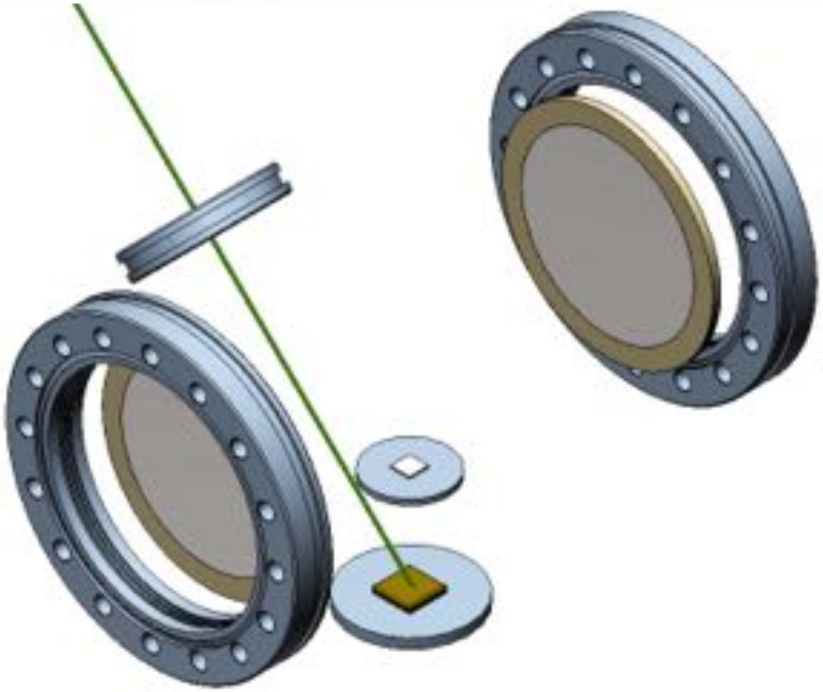
- chamber material: titanium
- Ultra High Vacuum chamber;
- Be-dome for *in situ* X-Ray experiments;
- adjustable leaks to 1×10^{-10} Torr l/sec

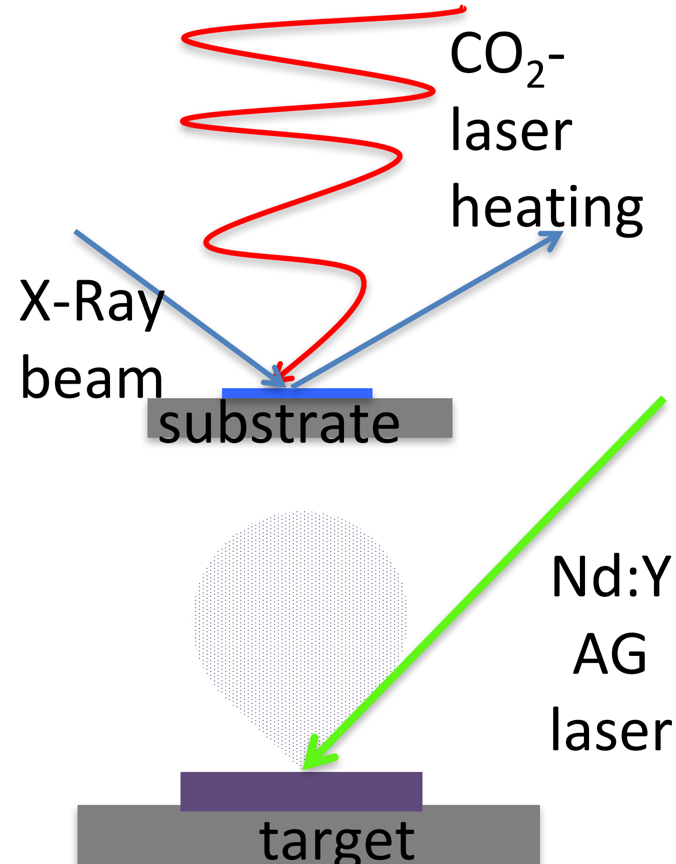
- the sample is displaced from 1-deposition to 2- X-Ray investigation positions

by Ksenia Maksimova

Portable PLD chamber: design III

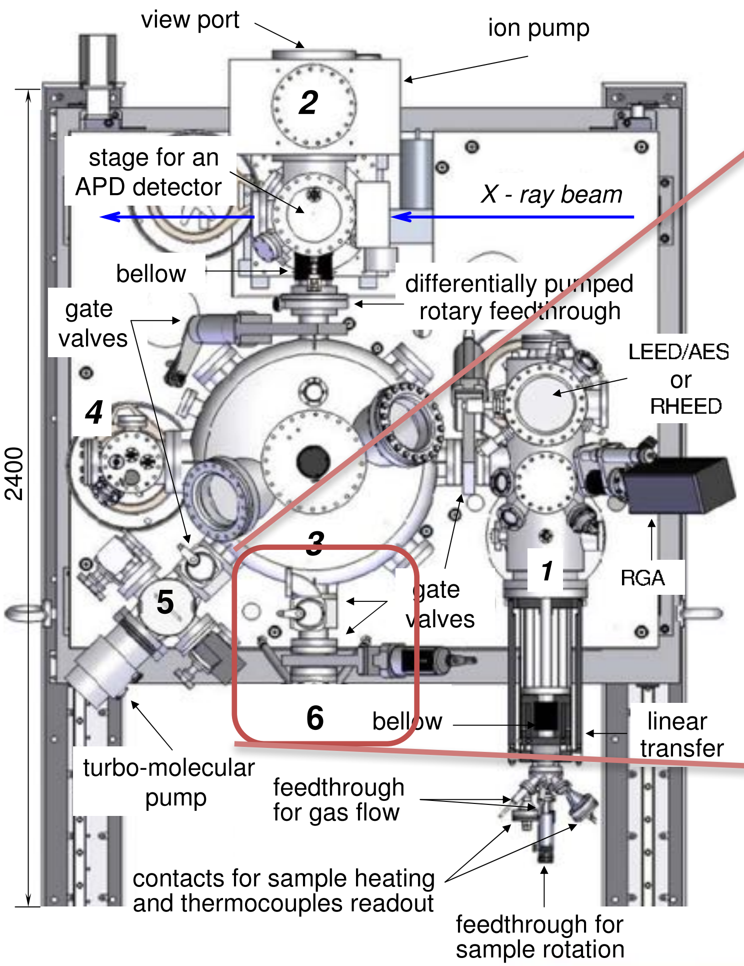
“Shadow” thin film deposition:

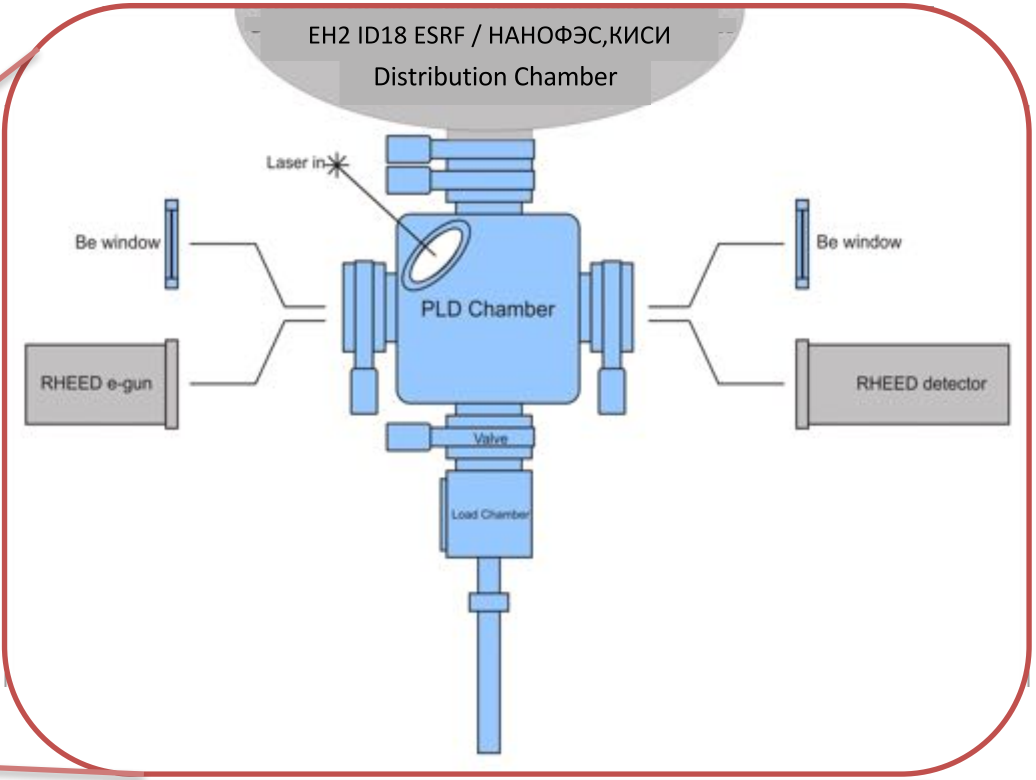




- Very slow growth speed;
- Absence macroparticles;
- Possibility doing X-Ray investigation during growth

Мобильные ростовые установки: ESRF ID 18 Nuclear Resonance PLD growth concept





- Исследование «чистых» металлов
- Возможность послойного формирования изотопических слоев Fe^{57}, Sn^{119} и т.д

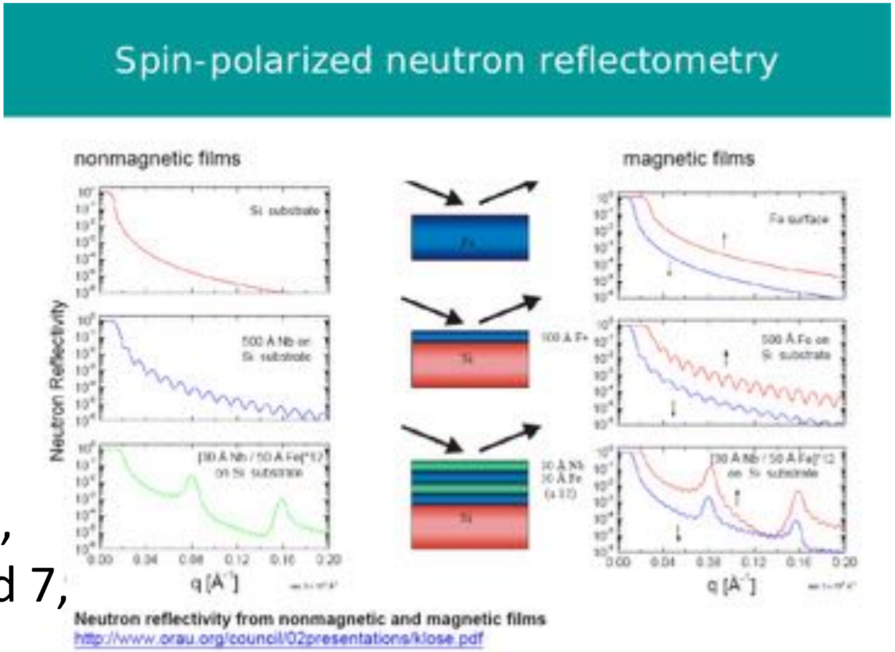
Структура доклада

- НОЦ «Функциональные наноматериалы» БФУ им. И. Канта
  - Возможности, задачи, установки
- Мобильные ростовые установки
  - Импульсное лазерное осаждение (PLD)
  - PLD *in situ* HAXPES @ P09 DESY
  - PLD chambers concept @ P23 DESY, ID18 ESRF
- In situ PLD установки на нейтронном источнике
  - PLD chamber concept @ MARIA, JCNS
  - Универсальная PLD установка для нескольких центров

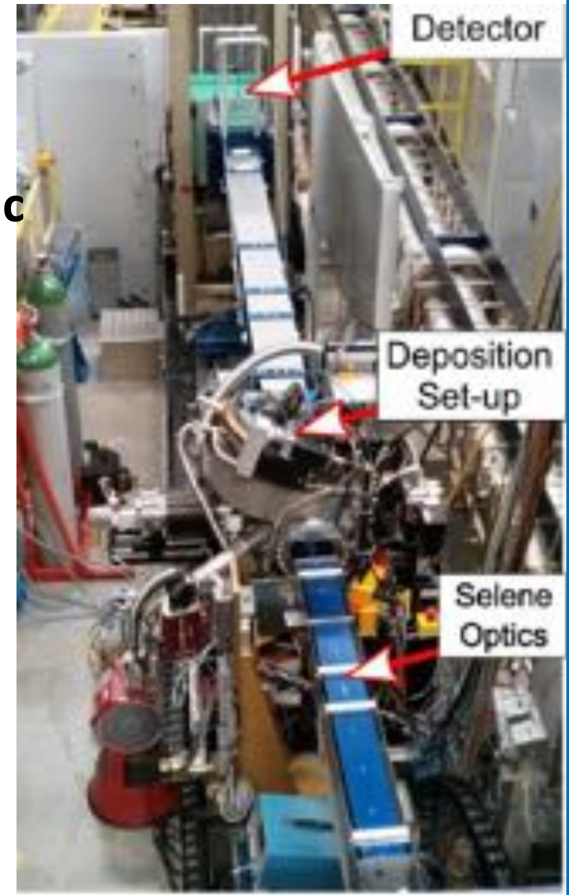
In-situ neutron investigations

✓**The aim – to investigate the properties of materials at early stages of thin films growth.**

Polarized Neutron Reflectometry (PNR) is very sensitive technique for structural and magnetic properties with atomic resolution.



W. Kreuzpaintner, Phys. Rev. Applied 7, 054004, 2017

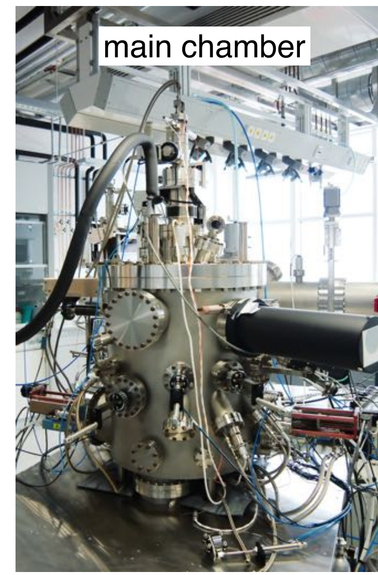


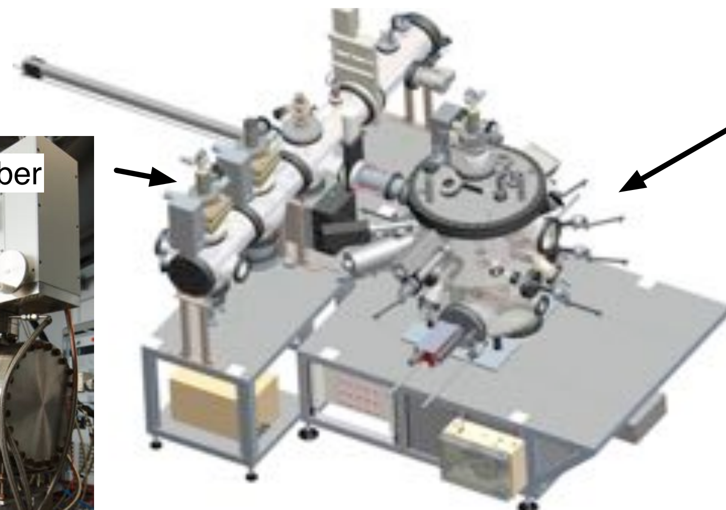
In-situ magnetron sputtering system at Swiss neutron spallation source SINQ

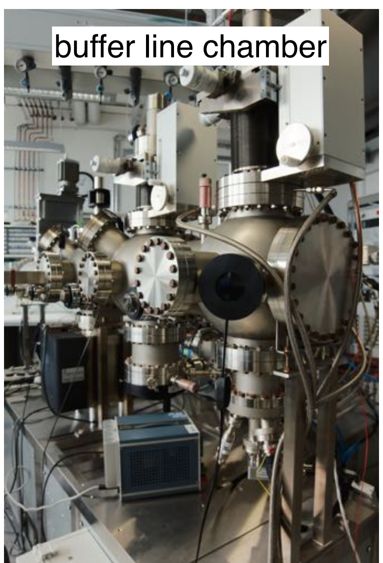
Molecular Beam Epitaxy setup

Base pressure < 10^{-10} mbar
Sources: 6 Effusion cells, 2 e-guns (each 4 crucibles), plasma source
Growth control via Quartz micro balances and Reflection High Energy Electron Diffraction (RHEED)
Substrate manipulator temperatures up to 1000 °C, sample size: up to Φ 2" and 20 mm x 20 mm
High reproducibility of sample growth:
Automated control of the growth procedure by "recipes" in the MBE system software
Supplied evaporation material:
Ag, Al, Au, Co, Cr, Cu, Fe, La, Mn, Ni, Nb, Pt, Sr, and Ti, other material on request

MBE: http://mlz-garching.de/mbe

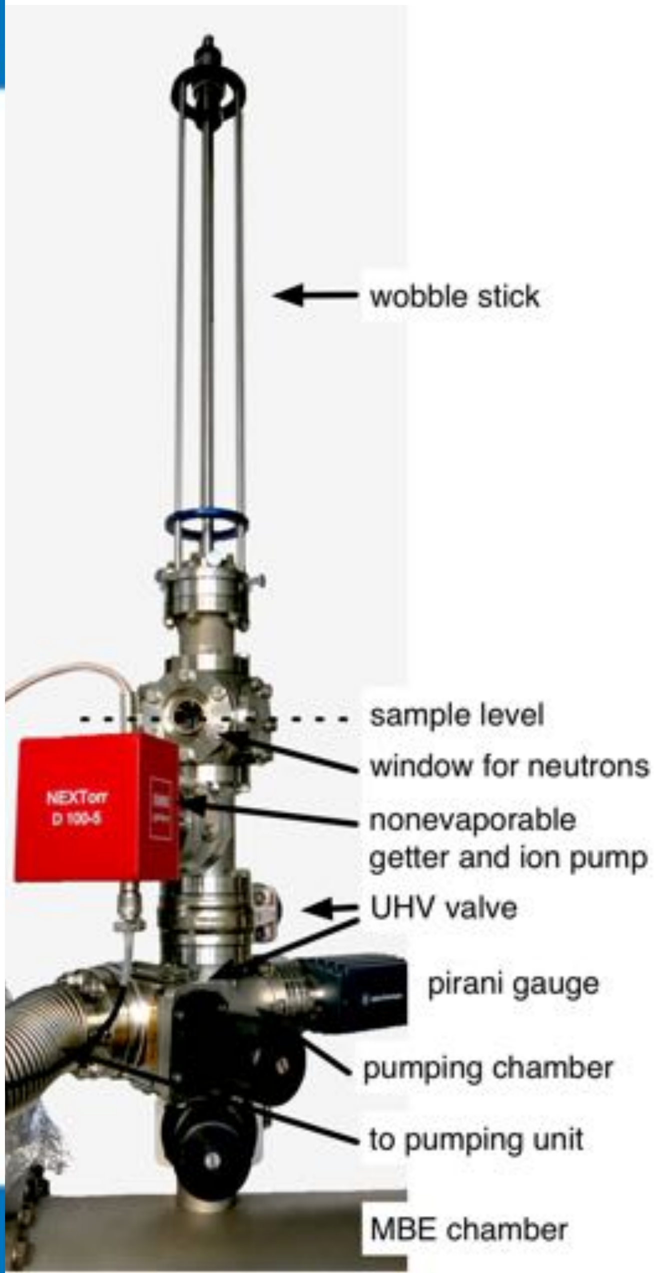






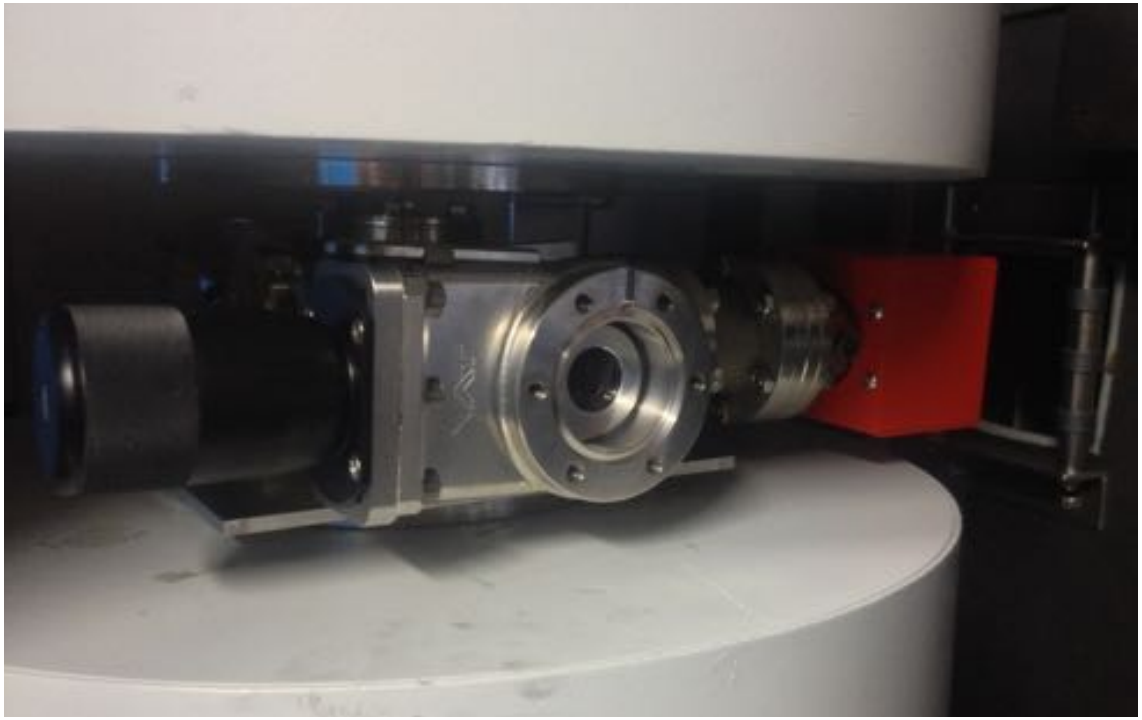
Buffer line chamber:
surface structure analysis via Low Energy Electron Diffraction (LEED)
chemical surface analysis via Auger Electron Spectroscopy (AES)
storage of up to 12 samples

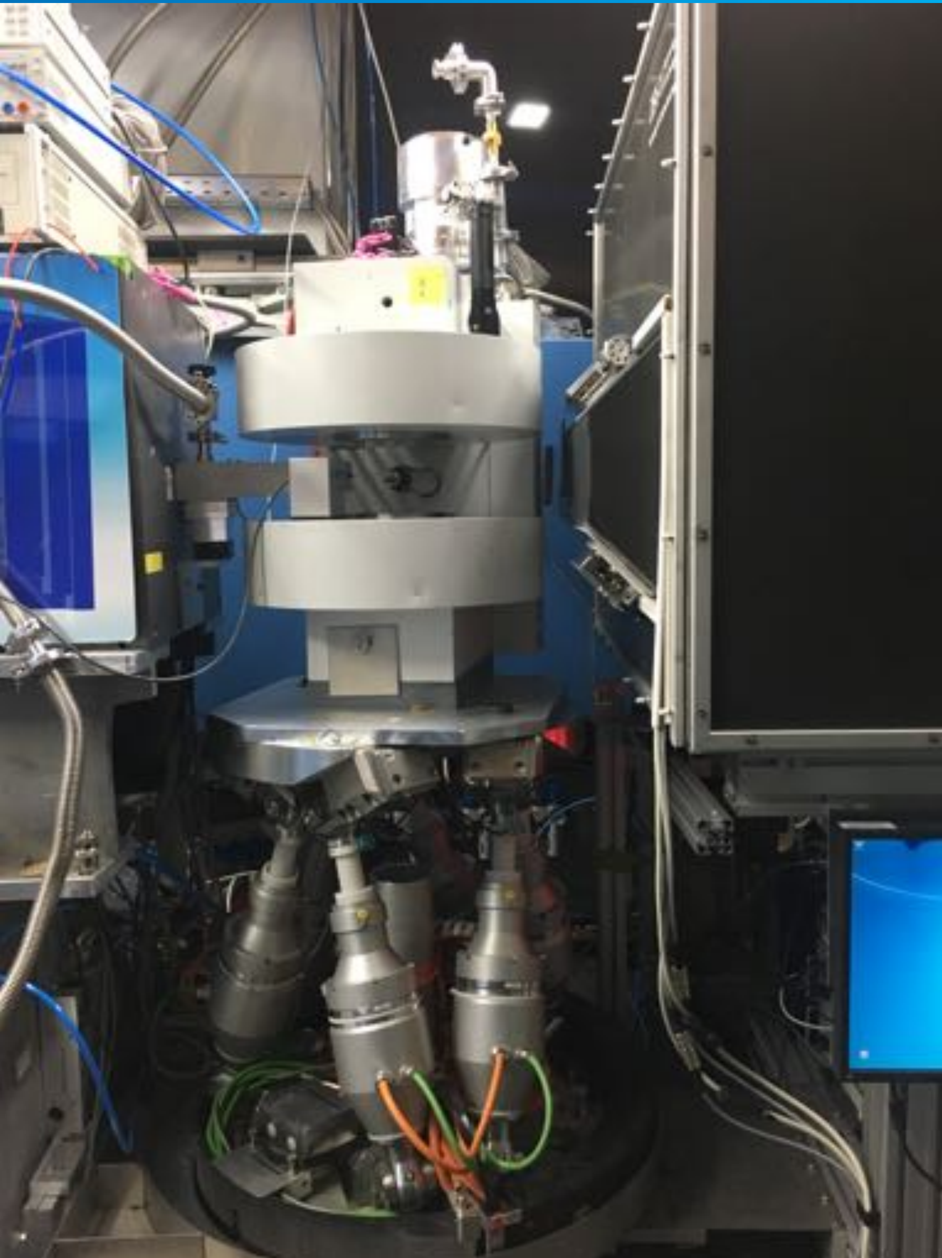
UHV-Transfer: quasi in-situ

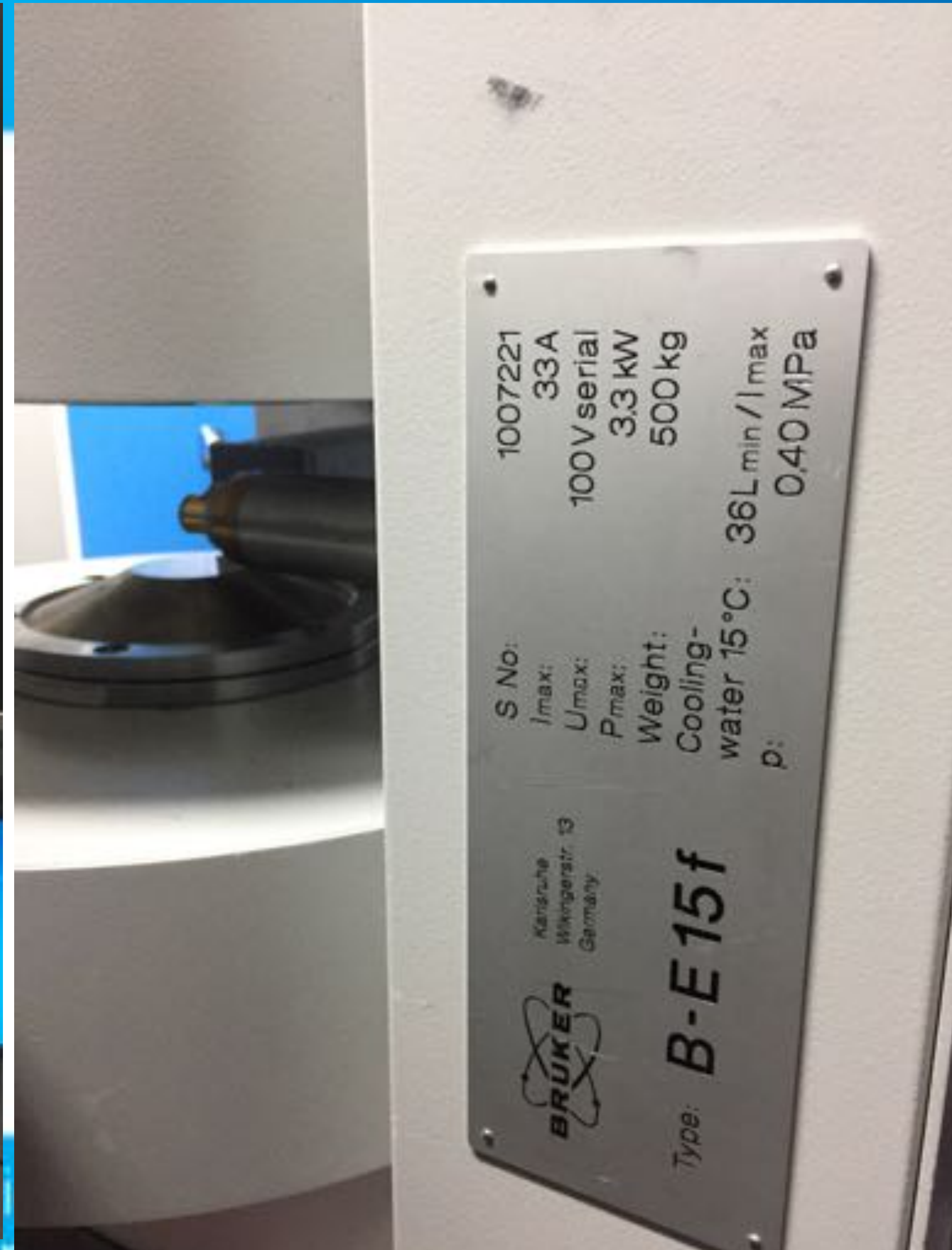


- DN CF-40 cube serves as main chamber
- two sapphire windows for the neutron beam
- a wobble stick, which serves also as a sample holder for samples of up to 1 cm2
- a DN CF-40 tee
- a nonevaporable getter and ion pump type Nextorr D 100-5 (SAES Getters SpA)
- DN CF-40 valve with window (for adjusting

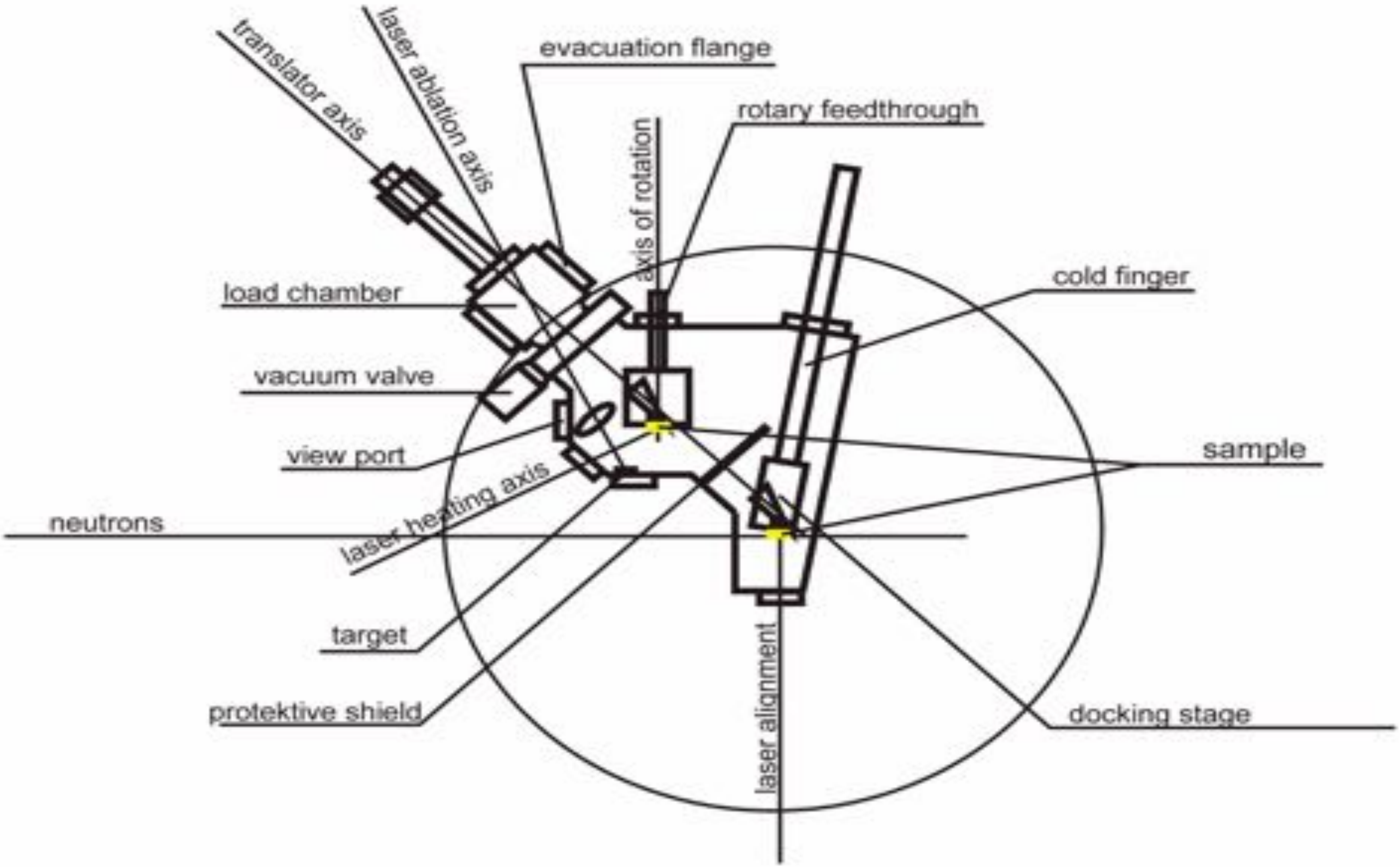
➜base pressure 2・10-10 mbar






BRUKER
Karlsruhe
Wikingerstr. 13
Germany
Type: B-E 15 f
S No: 1007221
Imax: 33 A
Umax: 100 V serial
Pmax: 3,3 kW
Weight: 500 kg
Cooling-water 15 °C: 36 Lmin / Imax
p: 0,40 MPa

In-situ neutron investigations @ MARIA



Методы

- Синхротронные методы:
  - Фото-электронная спектроскопия (РФЭС, HAXPES)
  - Синхротронная мессбауэровская спектроскопия
  - Дифрактометрия \ рефлектометрия

- Нейтронные методы:
  - Рефлектометрия поляризованных нейтронов (PNR)
  - Дифрактометрия

Спасибо за внимание!

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Уникальный идентификатор проекта RFMEFI58417X0031