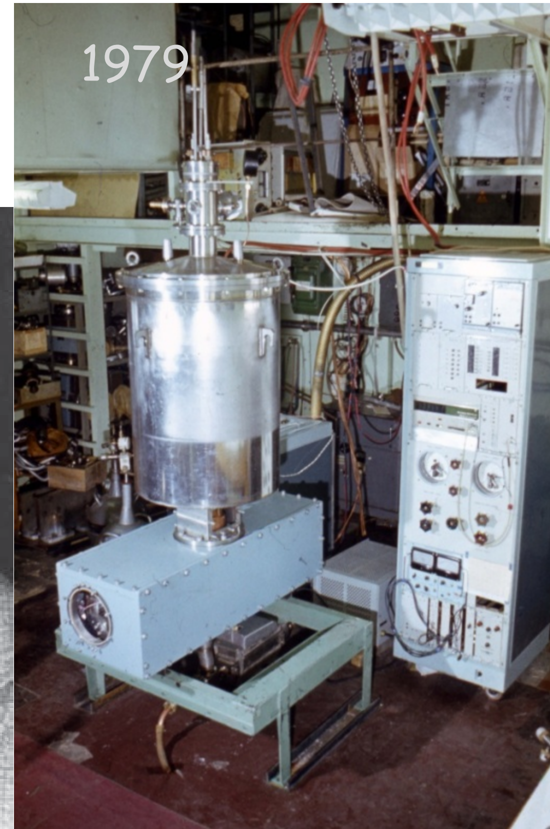
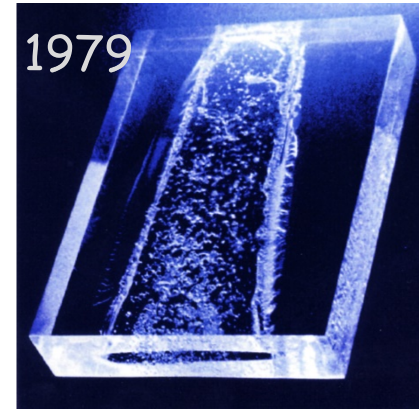
# SR activity in the Siberian Synchrotron and Terahertz radiation center

G.Kulipanov, E.Levichev, N.Mezentsev, V.Pindyurin, P.Piminov, B.Tolochko, *K.Zolotarev*, *BINP*

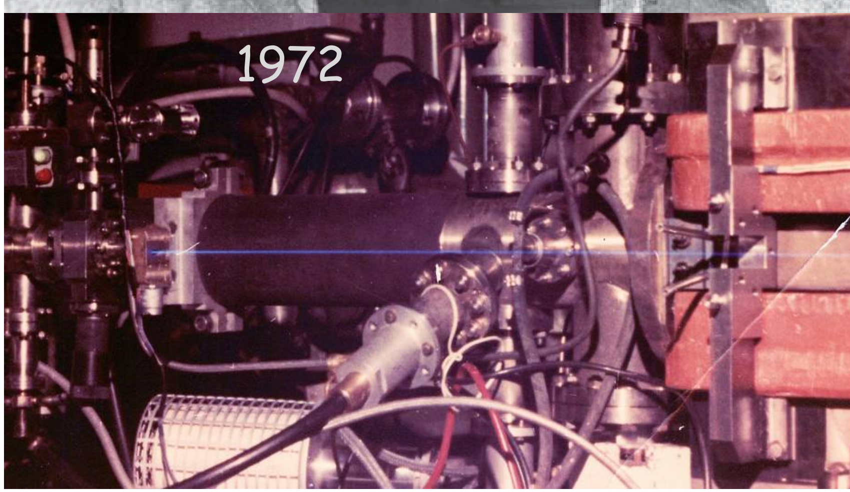
Synchrotron and Free electron laser Radiation: generation and application
SFR-2016
July 4-7, 2016
Budker Institute of Nuclear Physics
Novosibirsk

# SSTRC History

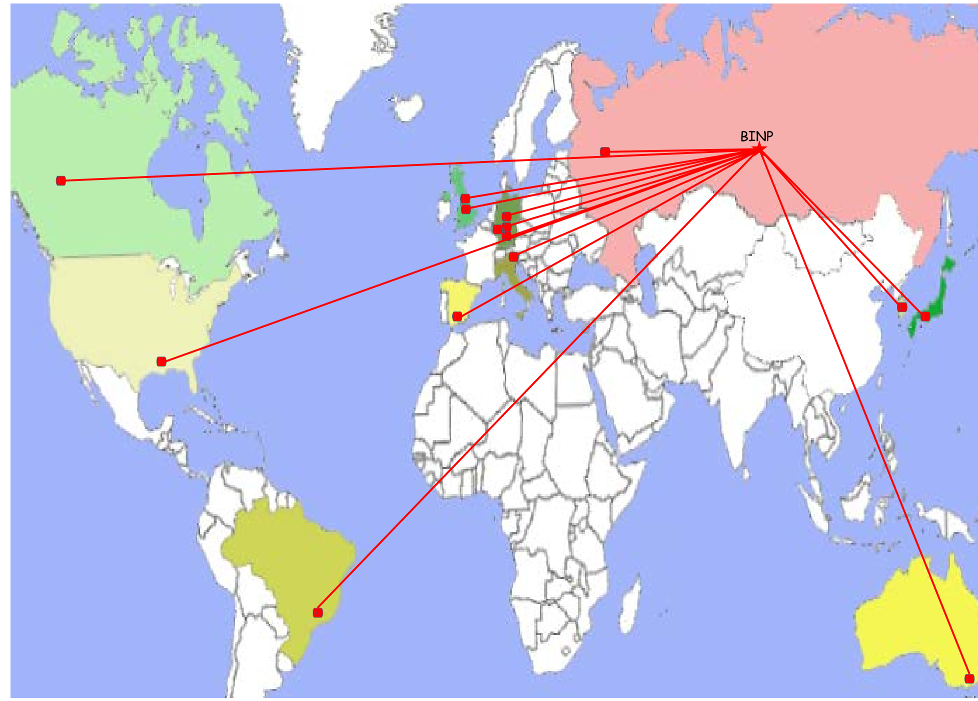







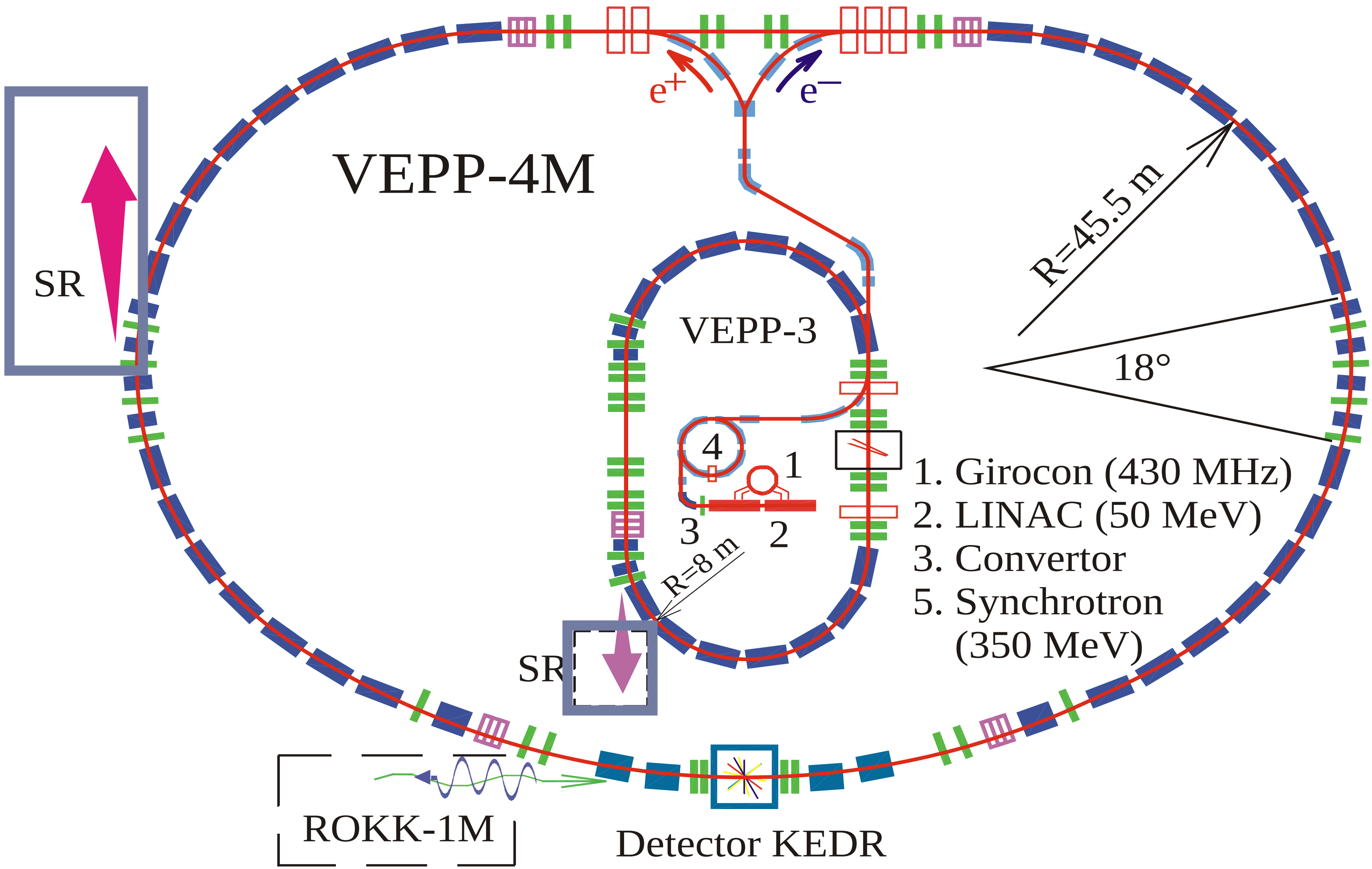

# SSTRC
# Main directions

- SR applications activity
- FEL developing, building, maintenance and upgrading
- FEL radiation applications in the teraherz rage
- Developing and fabrication superconducting insertion devices
- Developing and fabrication magnetic elements for accelerators
- Developing of the new light source for SSTRC
- SR and FEL conferences organization
- Education activity
- International collaborations



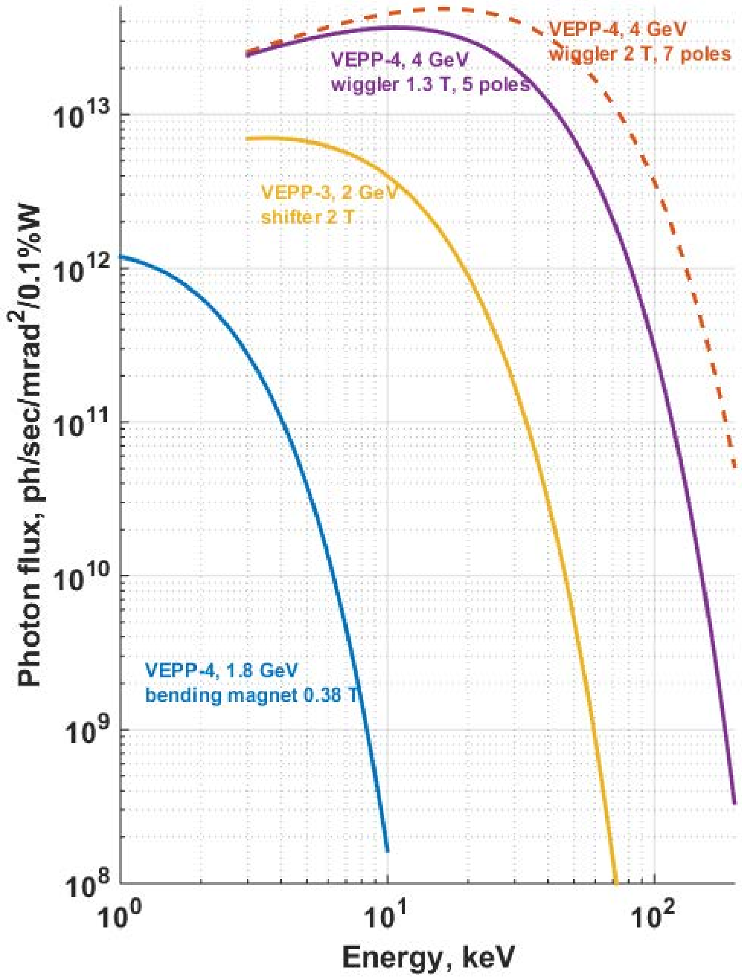



# Light sources in the SSRTC

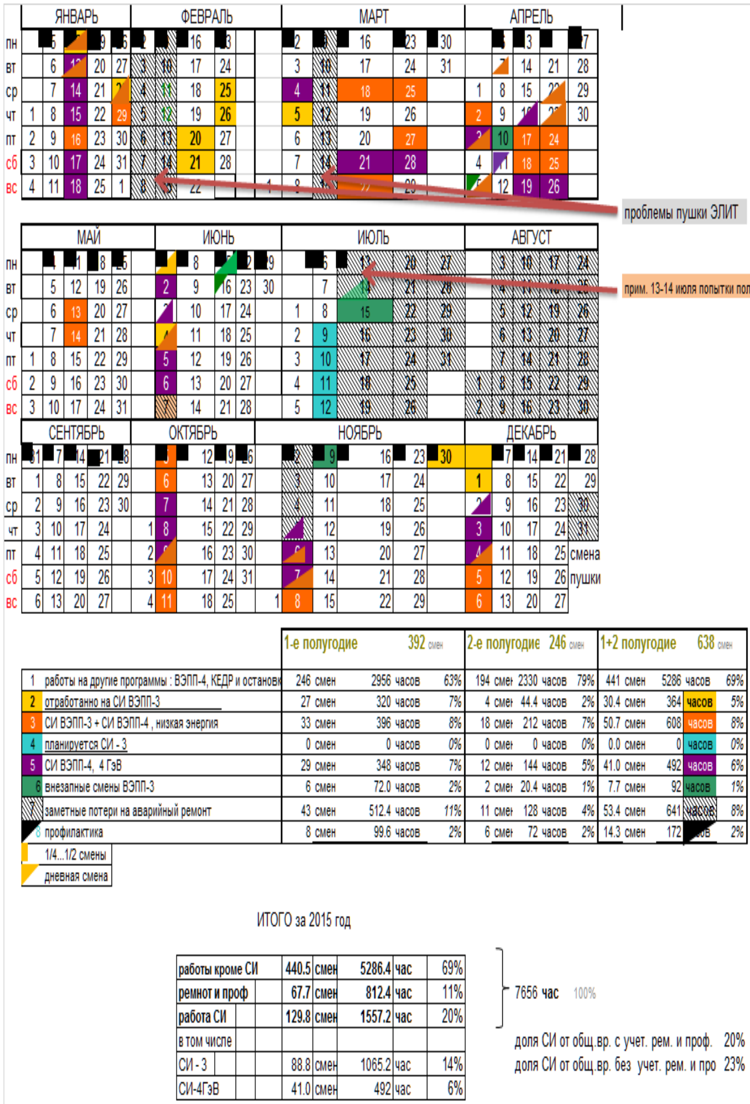


# SR sources

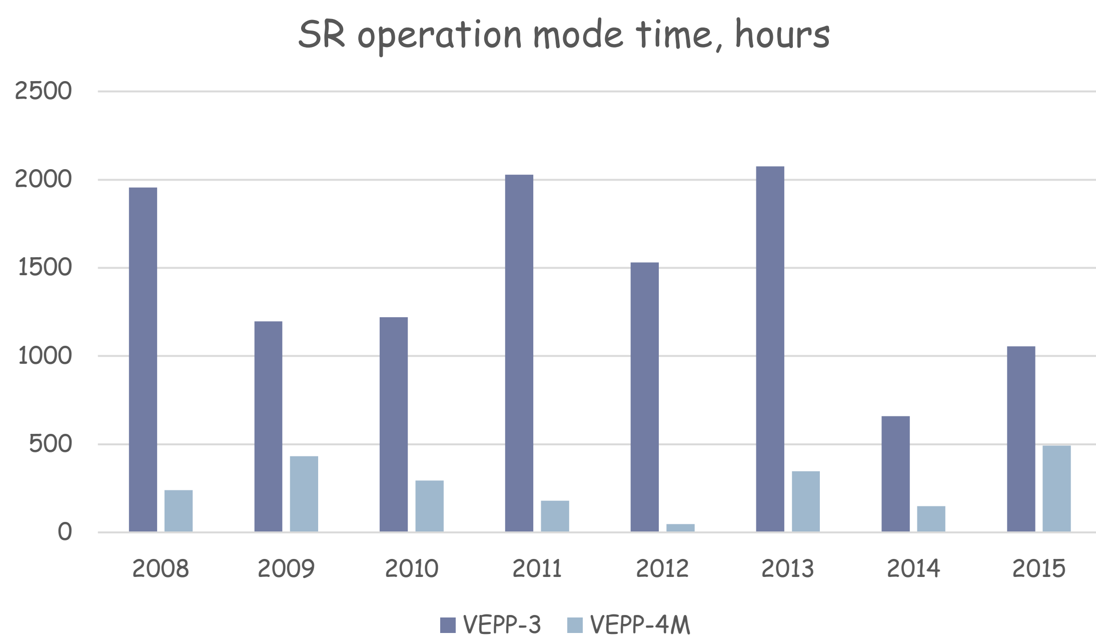
| | VEPP-3 | VEPP-4M Low Energy | VEPP-4M High Energy |
|---|---|---|---|
| Energy, GeV | 2 | 1.8 | 4 |
| Circumference, m | 72 | 366 | |
| Lattice type | FODO | FODO | |
| Emittance, nm rad | ~300 | 25 | 120 |
| Max. current, mA | 100 | 20 | 20 |
| Number of bunches | 1 - 2 | 1, 2, 4, 8 | 1, 2, 4, 8 |
| SR devises | Wave length shifter (2 T) | Bending magnet (0.38 T) | Multipole wiggler (1.3 T x 5 poles) |
| Optic function in irradiation point $\beta_x$, $\beta_y$, $\eta_x$, m | 2, 4.5, 0.7 | 9.64, 7.9, 0.9 | 9.7, 7.9, 1.16 |
| Source size in irradiation point $\sigma_x \times \sigma_y$ , mm | 0.9 x 0.3 | 2.3 x 0.1 | 1.5 x 0.25 |
| Critical energy, keV | 5.3 | 0.8 | 13.8 |
| Number of beamlines | 8 | 1 | 1 (3 stations) |
| | | | |



# Time for SR applications work (hours)

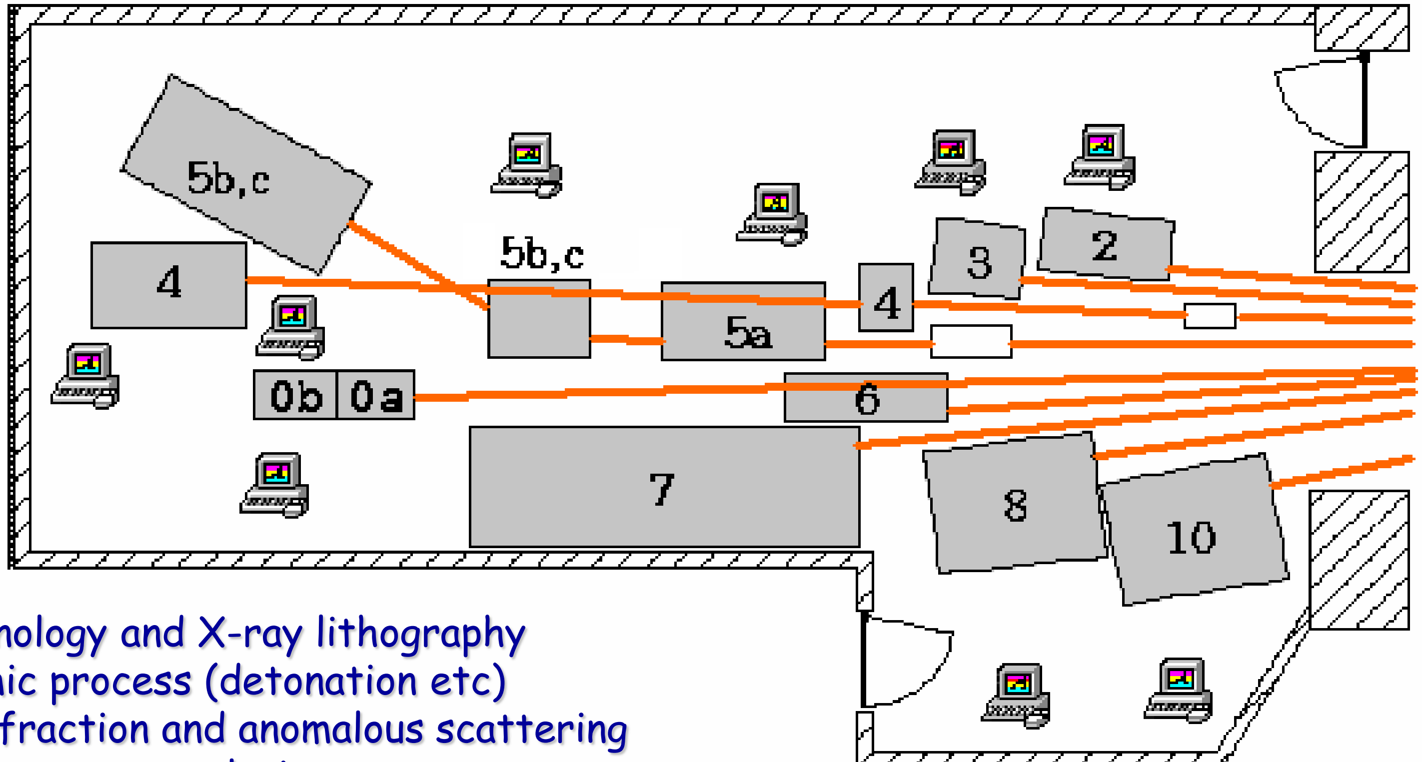



| Year / Light source | 2008 | 2009 | 2010 | 2011 | 2012 | 2013 | 2014 | 2015 |
|---|---|---|---|---|---|---|---|---|
| VEPP-3 | 1956 | 1197 | 1221 | 2028 | 1530 | 2076 | 660 | 1056 |
| VEPP-4M | 240 | 432 | 294 | 180 | 48 | 348 | 150 | 492 |



# VEPP-3 synchrotron radiation beamlines

VEPP-3 SR experimental hall (14x5 m)



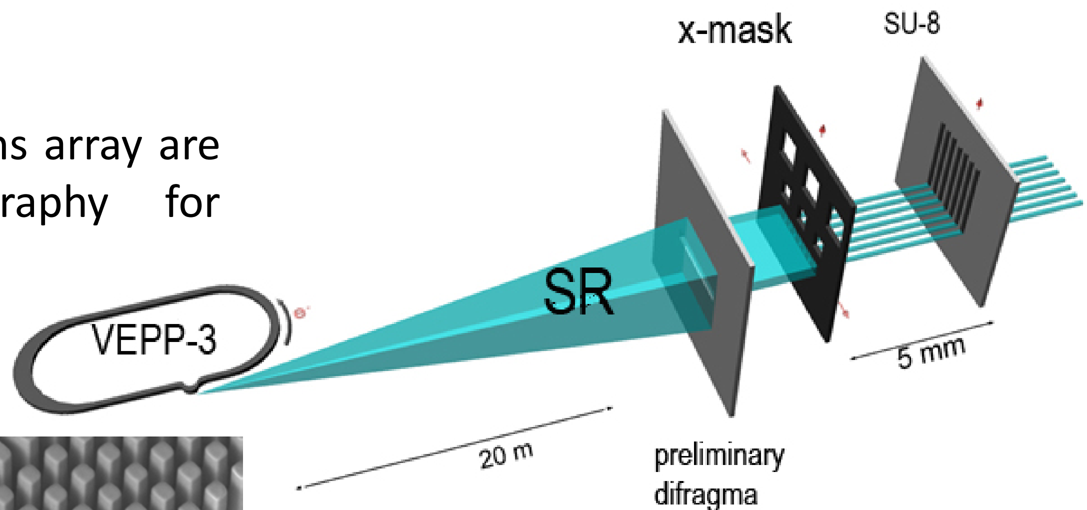

0a - LIGA-technology and X-ray lithography
0b - Fast dynamic process (detonation etc)
2 - Precise diffraction and anomalous scattering
3 - X-ray fluorescence analysis
4 - High pressure diffraction
5a – X-ray microscopy and microtomography
5b – Time resolved diffraction
5c – Small angle scattering
6a - Time resolved luminescence
6b – Precise diffraction-2
7 - SR monitoring station
8 - EXAFS-spectroscopy

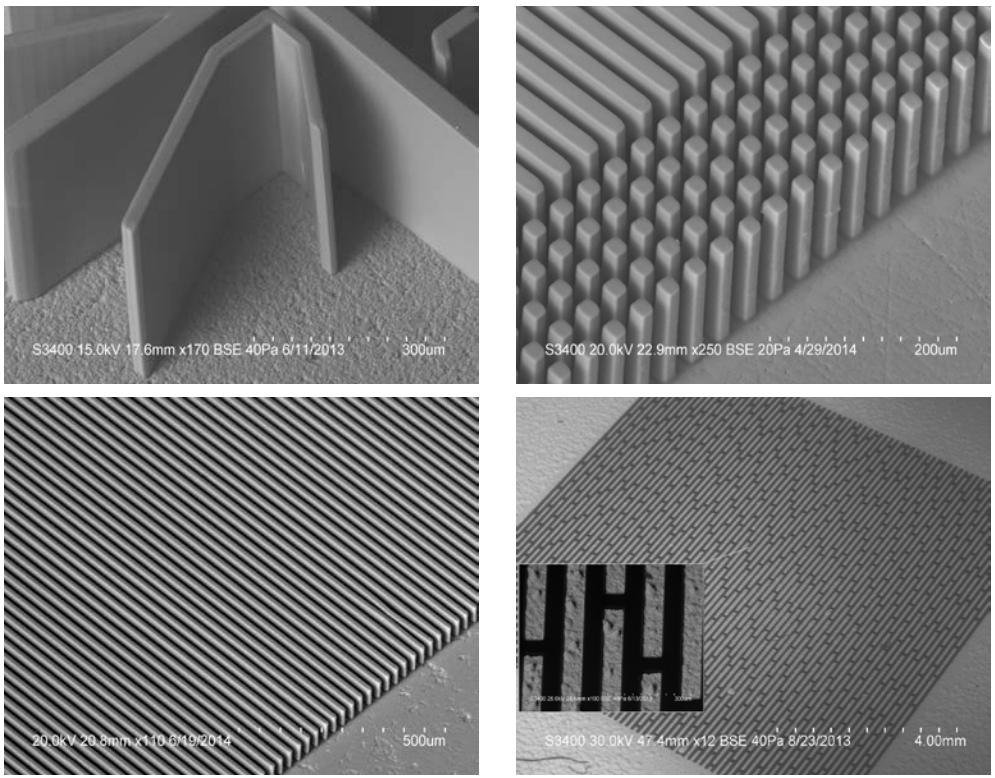
## VEPP-4 SR beamlines

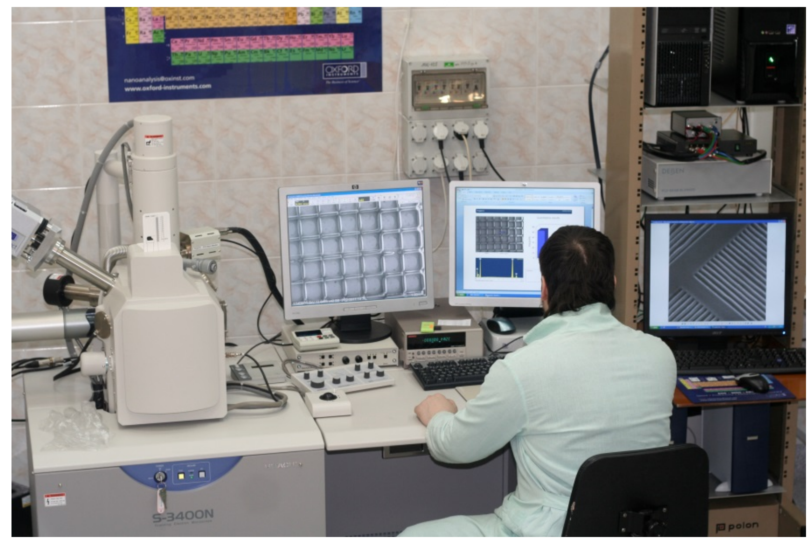
1. «Cosmos» (metrology in VUV and soft X-ray range 10-2000 eV)
2. Phase contrast microscopy, microtomography and hard X-ray fluorescence
3. «Vzryv-2» (nanosecond diagnostics)
4. «High presure» - assembling
5. "Plamya" beamline – developing
6. Precise difractometry (planning)

# "LIGA" station at VEPP-3

Single microbeam SR or microbeams array are used for Direct X-ray lithography for Fabrication of deep LIGA structures.



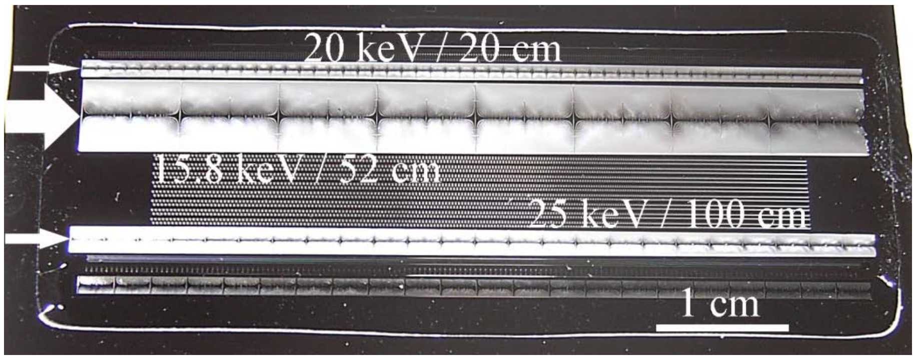



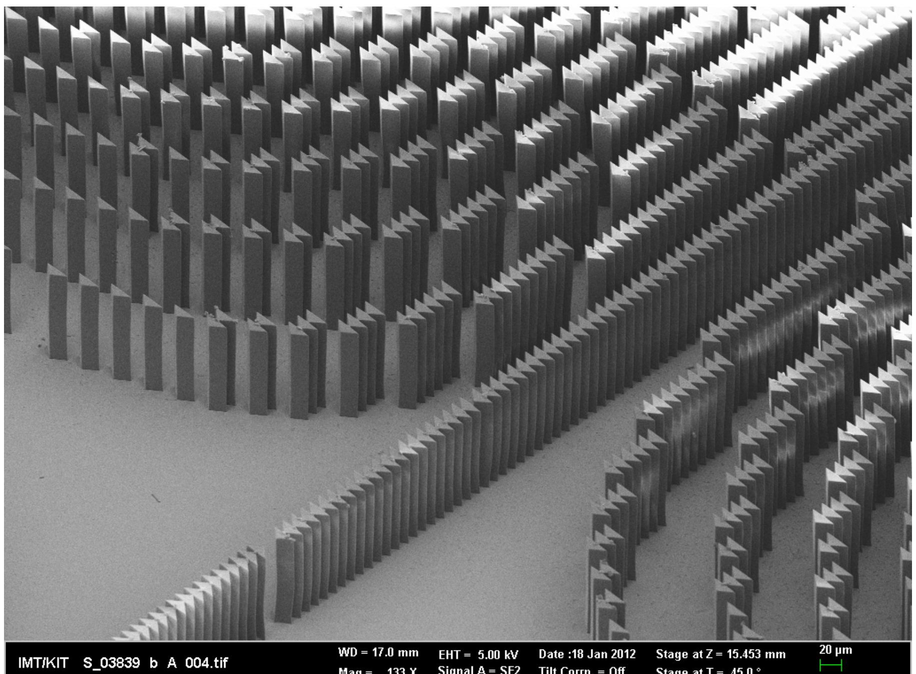



**Electron lithography.**
**SEM Hitachi Type II + Nanomaker** for microstructure forming in the thin PMMA layers (2-3 $\mu m$) for fabricating intermediate template for the soft X-ray lithography

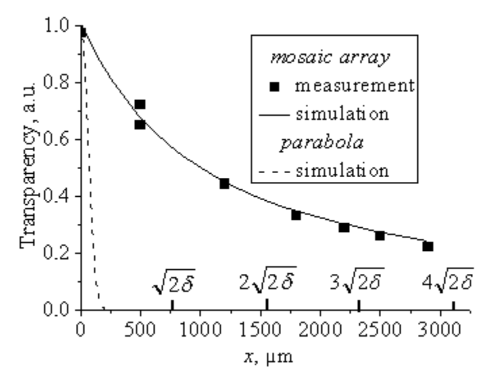
Samples of high aspect ratio microstructures: micro-lamellae, micro-grid, array columns

# Mosaic refractive lenses with big aperture




| E, | F, cm | *h*, µm | *l*, µm | *m* | *N* | Micro structures number, total | Aperture , µm | Calc.transparency |
|---|---|---|---|---|---|---|---|---|
| 20 keV | 20 | 15 | 26 | 54 | 43 | 102168 | 1290 | 0.40 |
| 15.8 keV | 52 | 12 | 31 | 8 | 242 | 470448 | 5820 | 0.29 |
| 15.8 keV | 52 | 12 | 31 | 8 | 121 | 118096 | 2904 | 0.44 |
| 25 keV | 100 | 20 | 41 | 22 | 33 | 24684 | 1320 | 0.62 |

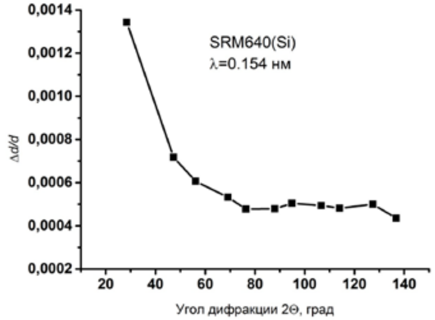


# Beamline 2 Anomalous scattering

The main parameters of the station
Monochromator:
"Channel cut» Si, c working plane (111)
Energy range: 7-20 keV
collimator:
Slits Ta
beam on the sample size 0.1 ÷ 2 × 5 mm2
The diffractometer:
scanning angle range 2Q = -10 ÷ 140 °
Minimum scanning step 0.001 °
Sample:
Flat washer ø30 mm or cylindrical capillary ø0.5 ÷ 1 mm
detection systems:
PMT scintillator NaI (Tl)
One-coordinate detector OD-3M-350

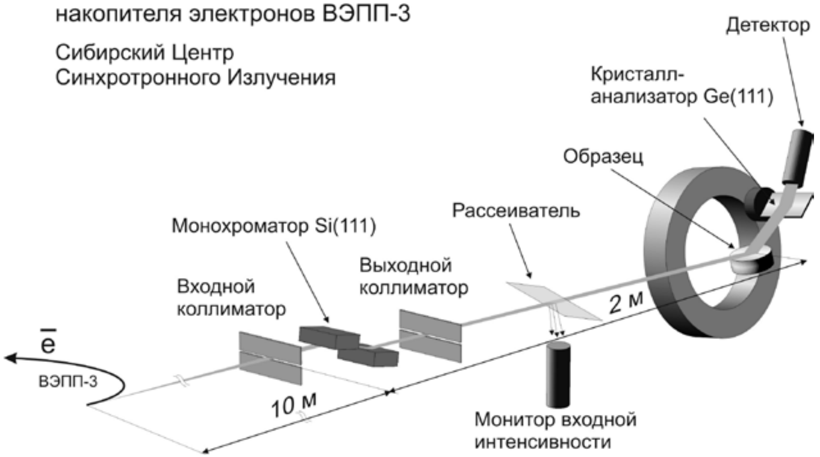
**Realized methods**

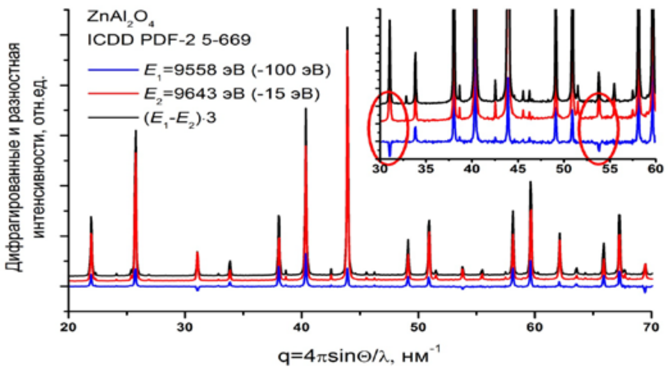
- diffraction with high angular resolution;
- diffraction in grazing incidence (Grazing Incidence Diffraction);
- small-angle diffraction (diffraction on long-structures);
- obtain the radial distribution of the electron density functions;
- Diffraction using the resonance scattering




Канал СИ №2
накопителя электронов ВЭПП-3

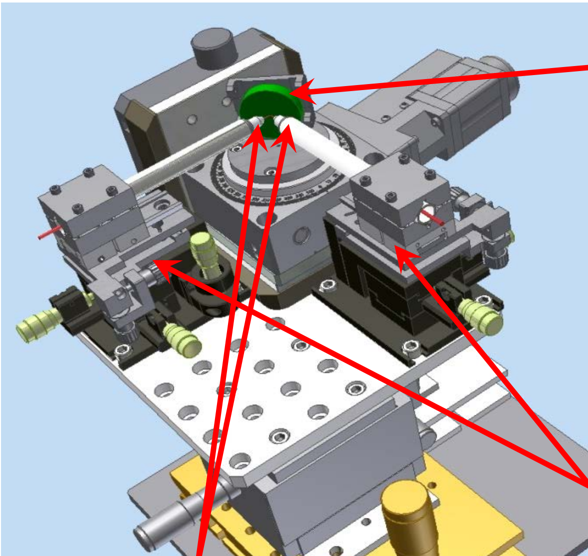
Сибирский Центр
Синхротронного Излучения




Resonant scattering near the absorption Zn K-edge. Model object - zinc-aluminum spinel. Record the contrast values of the diffracted intensity at different radiation energies.

# Beamline 3, Scanning $\mu$SRXRF
# Confocal polycapillary X-ray optics

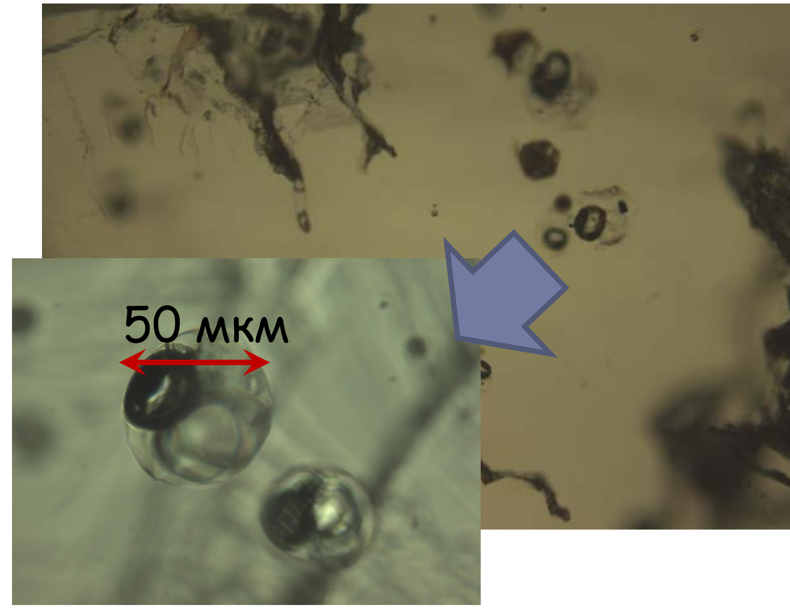


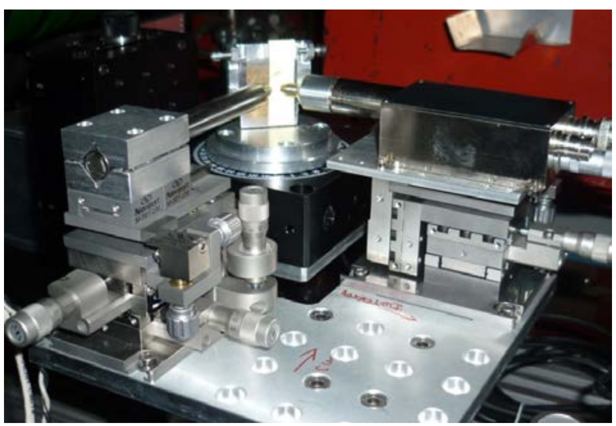
sample

Lenses adjustment stages

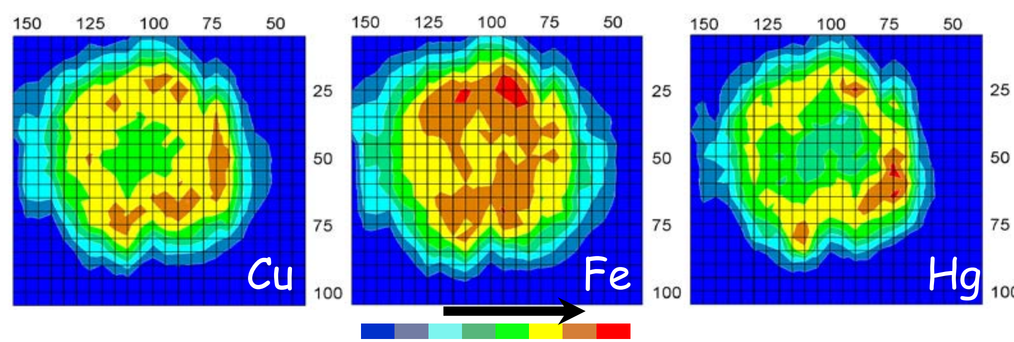
X-ray polycapillary lenses

Microfluid insertions



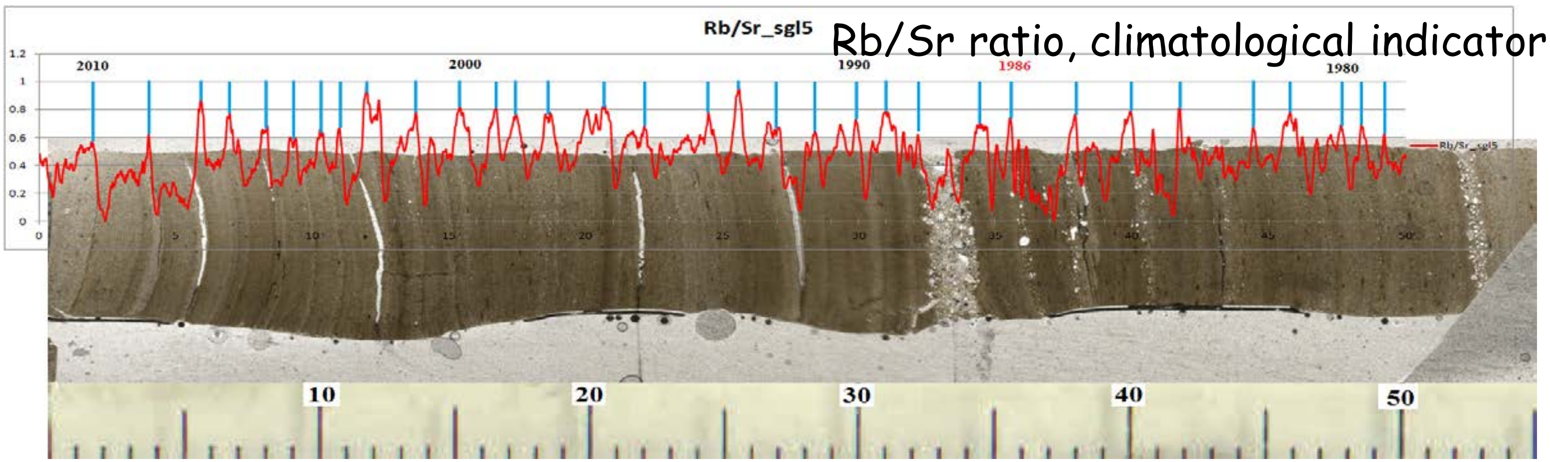



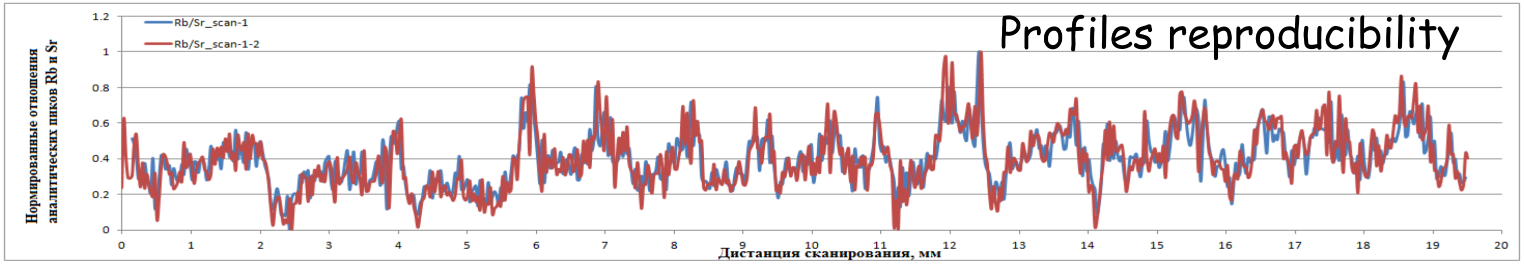
Spatial resolution about 10 $\mu$m
3d reconstruction

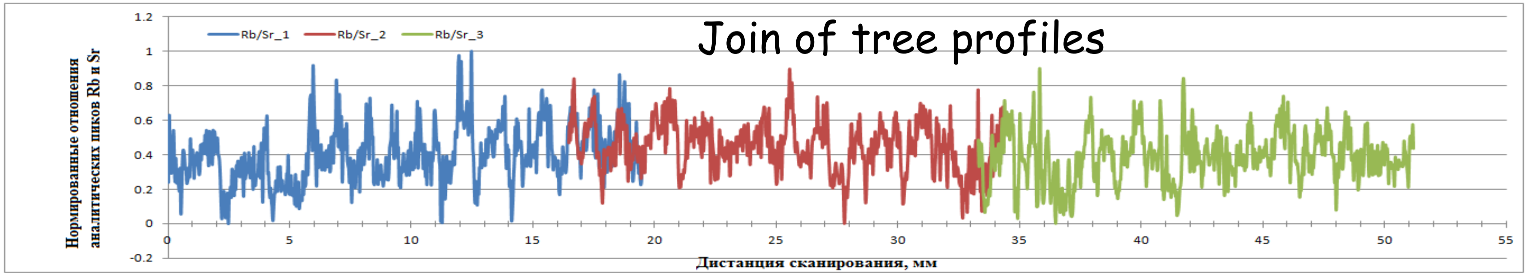



Elements distribution in the coss-section of the human hair

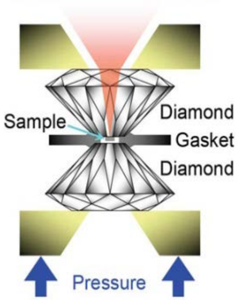
# Beamlibe 3. SRXRF. Lake bottom sediments analysis.

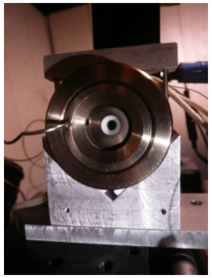


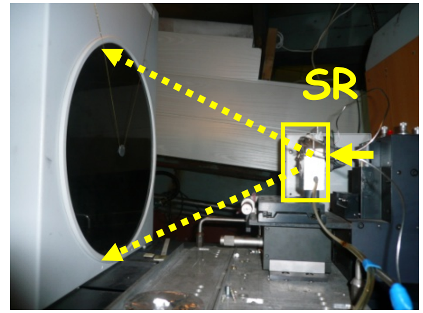



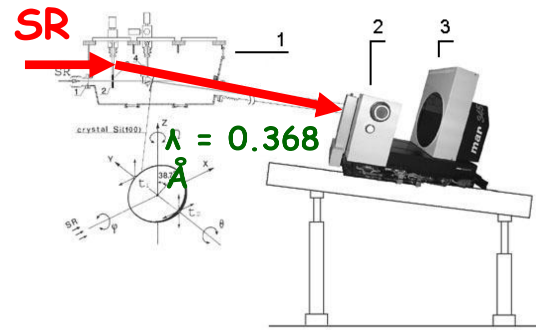



# Equipment for XRD experiments with high pressure and high temperatute
# Beamline 4, VEPP-3



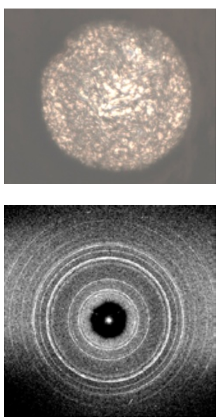


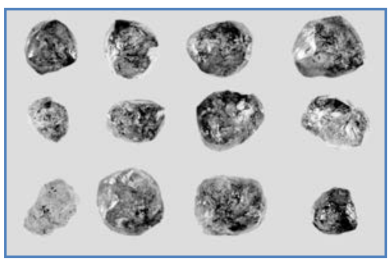

## High pressure diamond anvil cell and general view for the diffraction experiment

## Stability of hydrocarbon compounds at high pressures and temperatures and implications for the deep structure of the Earth and planets

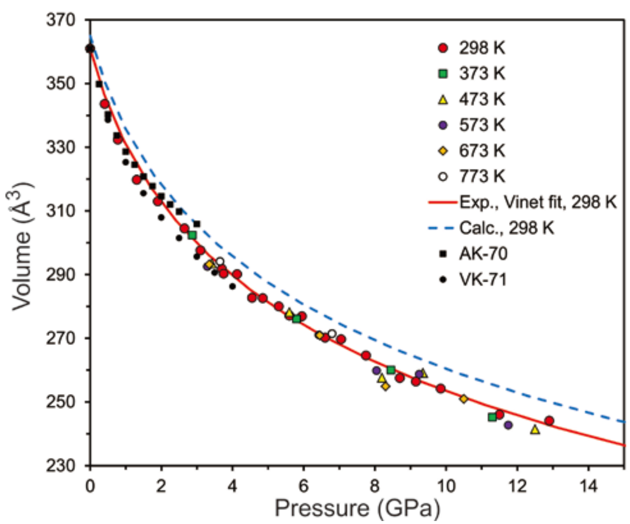
**polycyclic aromatic hydrocarbons - important components of inclusions in the deep minerals and meteorites**



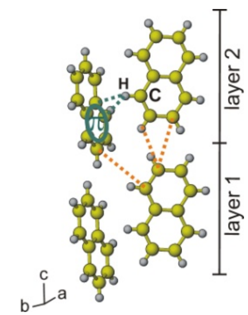
View in the chamber and the paten of powder sample at P ≈ 3 GPa

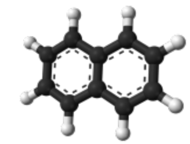


polyphase inclusions hydrocarbons in diamonds from deposits north-east Siberian platform (Томиленко и др., 2001, Доклады РАН).



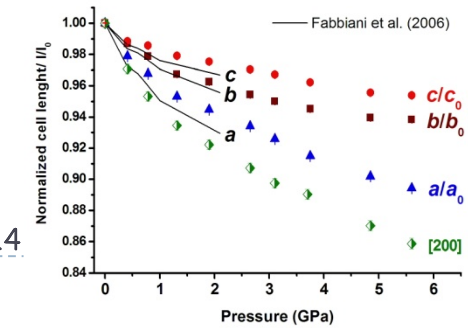

Pressure dependence of the unit cell volume of naphthalene $C_{10}H_8$ at 298-773 K.



molecule naphthalene

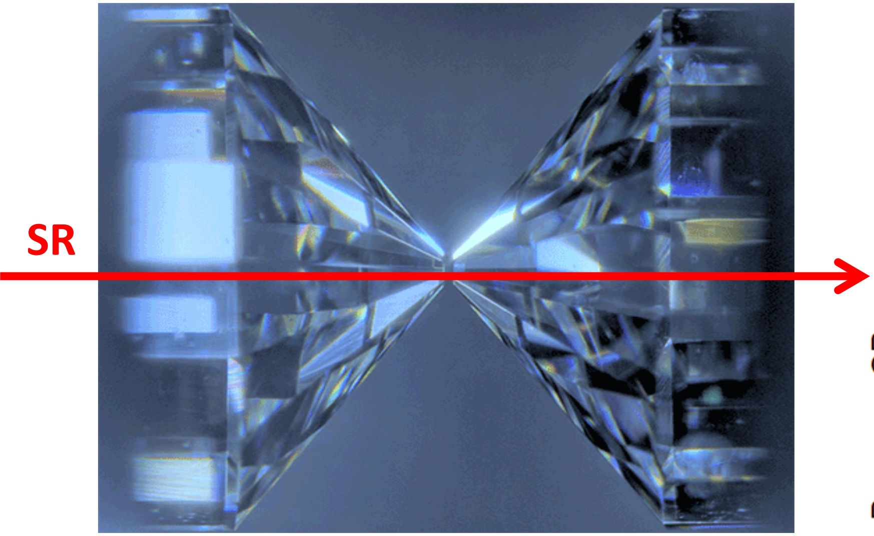
Structure and anisotropic compressibility naphthalene 0-6 GPa



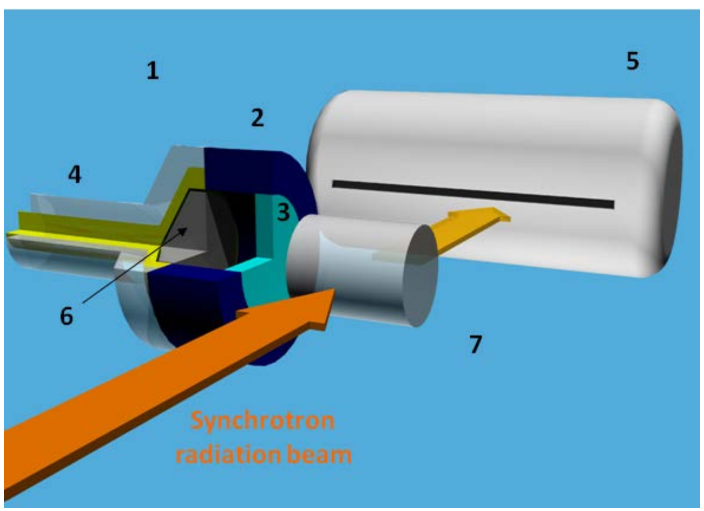

**Murchison meteorite**

Aromatic hydrocarbons predominate in hydrocarbon matter of meteorites *(Pering, 1971, Science*

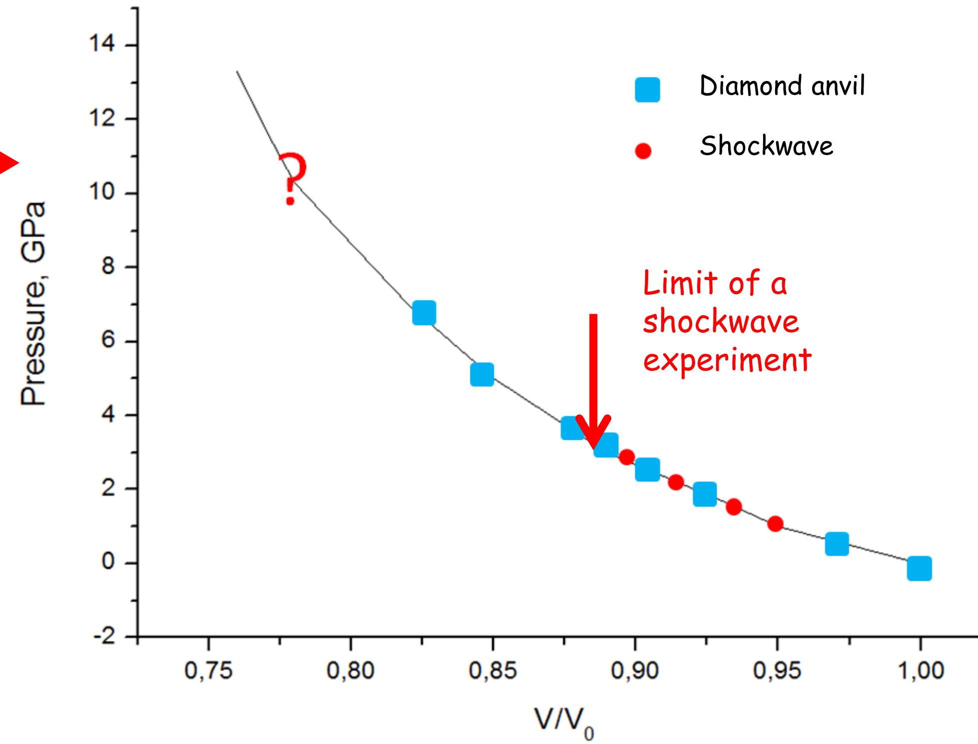
# The equation of state of explosives




Diamond anvil



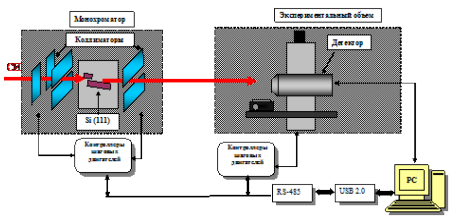

Shockwave experiment

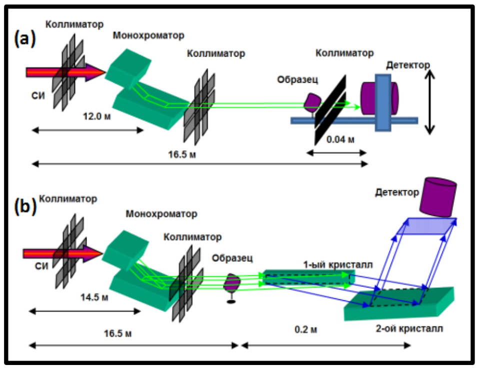



Experimentally obtained curve equation of state of shockwave loading TATB and compression in the diamond anvils

# Beamline 5a. X-ray microscopy and microtomograpy

Main layout

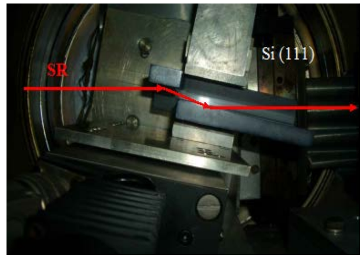





Imaging schemes
(a) - without magnification, (b) - scheme using asymmetrically cut crystals

**The main parameters of the station**
Monochromator:
Two crystals, silicon, (+ n, -n) c working crystallographic plane (111)
The range of photon energies of monochromatic radiation: 5-45 keV
Spatial resolution
In the circuit without increasing: 50 μm
In the circuit with increasing 2 μm

Monochromator



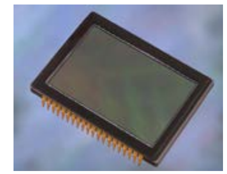

Channel cut monochromator: Si (111)

Experimental hatch

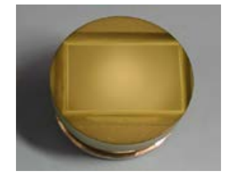

1 - sample 2 - first asymmetrical crystal, 3 - second asymmetric crystal, 4 - Detector

Two dimensions detector“Photonic Science”

Effective range: 62 x 41 mm2
The scintillator: Gadolinium oxysulfide
Energy range: optimum 5 - 35 keV
Range of registration: 65536 (16-bit)

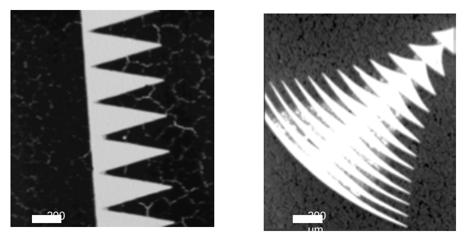


CCD 4008 x 2670
pixel size 9x9 μm$^2$

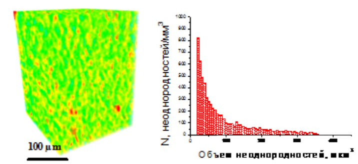


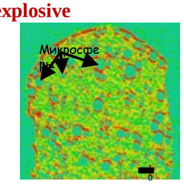
Fiber optics with magnification 1.73

**Testing of the X-ray transparent coatings**

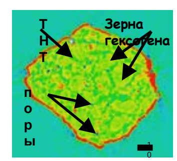


**Density distribution in the explosive**



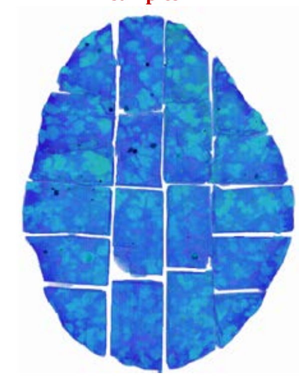
Эмульсионное ВВ



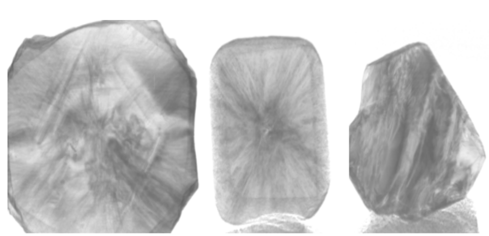

TNT and hexogen mixture

Hexogen with 7% TЙ

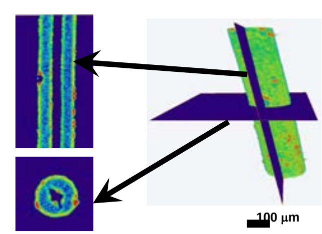
**Minerals distribution in geological samples**



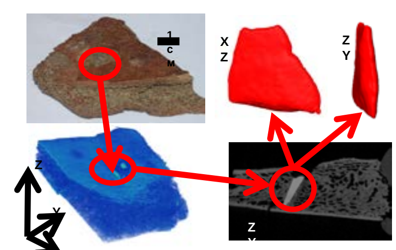
**X-ray topography on natural diamonds**



**Archaeological research**




Hair from accent barrows

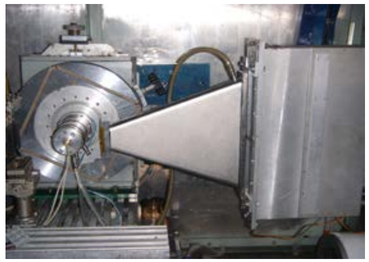


Detail of the tip in buffalo bones

# Beamline 5b. Diffraction movie

One dimension detector OD-3
WAD mode

One dimension detector OD-3
SAX mode



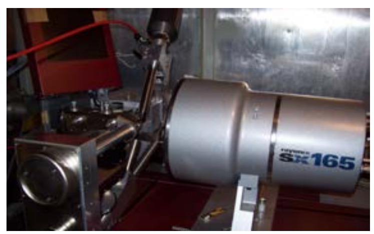
**Dedication**
Research of the phase and structural transformation during chemical reaction by using X-ray diffractometry

Main parameters:
Monochromator: cut silicon single crystal Si (111)
Energy: ~ 8.2 keV

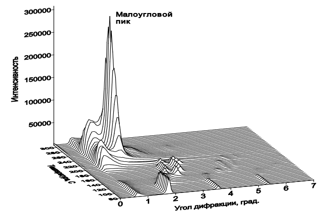
**One-coordinate detector OD-3:**
Angle range: ~ 30 degrees (at a distance of up to 350 mm of the sample)
Channel: ~ 0.01 °
The number of channels: 3328
Minimum time frame - 1 ms
Maximum load: 10 MHz

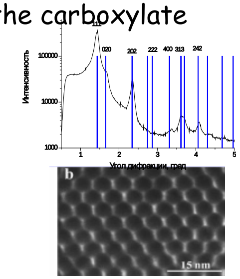
**XY detector MarCCD**
Pixel Size - 80 * 80 mm
The diameter of the working area - 165 mm
The minimum reading time – 2 sec

two dimensions detector MarCCD



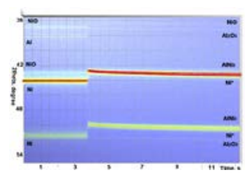

Formation 3D-structure of the silver nanoparticles produced by the decomposition of the carboxylate



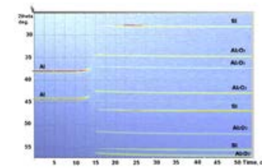



Phase transformation dynamics in the Self-propagating high temperature synthesis (SHS) in the mechanical composite materials

$NiO + Al + Ni – AlNi_3$

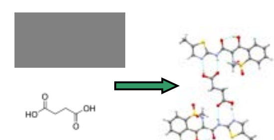


$SiO_2 + Al \rightarrow Si + Al_2O_3$

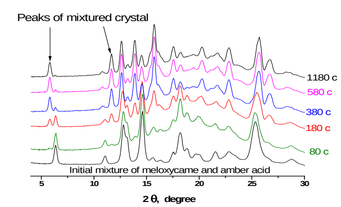


… and during formation mixed organic crystals

Meloxycame

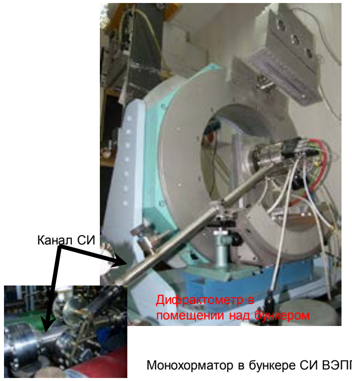


Amber acid



# Beamline 6. Precise diffractometry

## General view

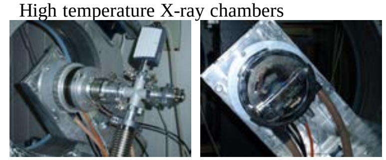



Монохорматор в бункере СИ ВЭПП-3

## Layout


## Equpment

High temperature X-ray chambers

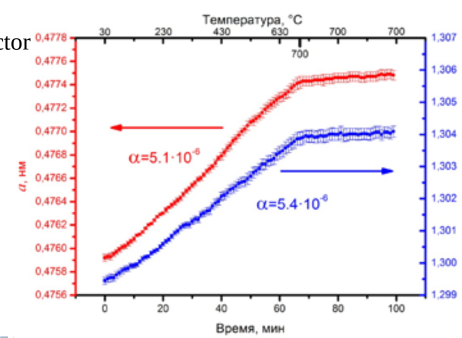


Luggage Anton Paar HTK-2000 experiments at temperatures up to 1400 ° C in air or an inert atmosphere to 2000 ° C in vacuum.

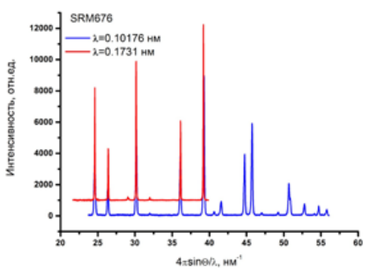
Camera Anton Paar XRK-900 experiments at temperatures up to 900 ° C in an oxidizing or reducing environment, and gas mixture pressure of from 0.1 mbar to 10 bar.

- 3-channel system of preparation of gas mixtures on the basis of mass flow-controllers;
- hydrogen generator
- Gas analyzer based on SRS RGA-100 quadrupole mass spectrometer

Corundum lattice parameter change due to thermal expansion by heating in an inert atmosphere. Camera XRK-900, envinment - He.




The main parameters of the station
Monochromator:
A single-crystal, with the beam deflection in the vertical plane at an angle ofapproximately 30 °;
Crystals: Ge (111), Si (111), Si (220);
The discrete set-energy radiation:
7.162 keV, 7.460 keV, and 12,183 keV
collimator:
Slits output;
beam on the sample size 0.5 × 5 mm2
detection systems:
One-coordinate detector OD-3M-350;
The range of angles 30 °,
resolution 0.01 °, the time resolution of 1 ms.
Sample Holders:
High-temperature X-ray cameras Anton Paar XRK-900 and HTK-2000



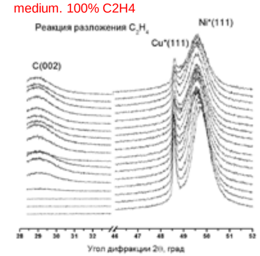

XRD patterns of corundum, obtained at different photon energies in a fixed detector position

### Realized methods

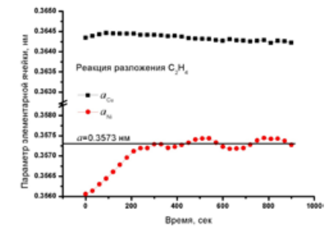
diffractometry with time resolution at high temperatures (up to 1400 ° C in air to 2000 ° C in vacuum);
diffractometry with time resolution in a reaction medium (up to 900 ° C at gas pressures from 0.1 mbar to 10 bar);

## Phase composition of Ni-Cu catalysts for the synthesis of nitrogen-containing carbon nanofibers and its changes in response

Changing the state of the catalyst in a reaction medium. 100% C2H4

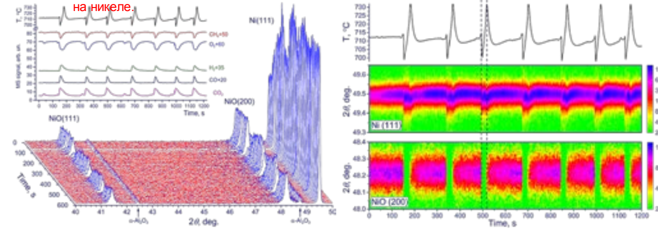



Changes in the catalyst lattice parameter in a reaction medium. 100% C2H4



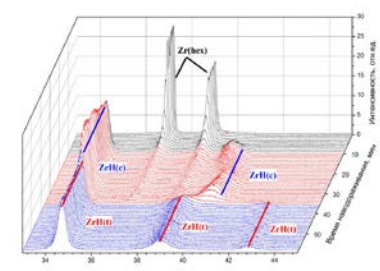

## Autooscillations reaction rate in the catalytic oxidation of light hydrocarbons to Ni and Pd

Изменение фазового состава катализатора, состава газовой смеси на выходе из реактора и температуры катализатора в процессе каталитического окисления метана на никеле.



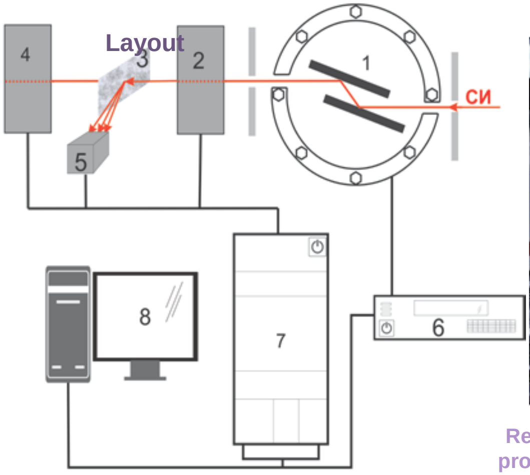

## In Situ Investigation of the structure changes alloy based on zirconium with saturated hydrogen from the gas phase

In an atmosphere of hydrogen at 350 ° C is formed of a cubic phase of zirconium hydride, and at 450 ° C there is a transition to the tetragonal phase cubic.



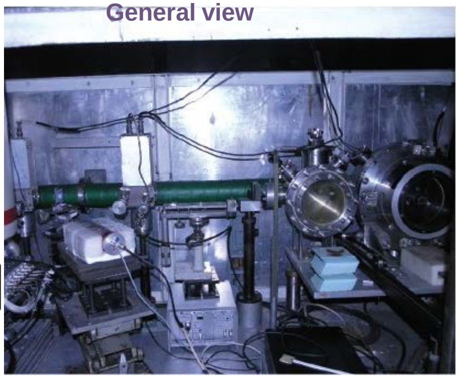
# Beamline 8 XAFS

## Layout




1 - two-crystal monochromator; 2.4 - ionization chambers; 3 - the sample; 5 - the detector (PMT / PDP); 6 - the controller; 7 - management and recording system; 8 - PC

## General view



## Purposes

Carrying XANES and EXAFS researches - determination the charge states of the elements and structure of the local agents in various states of aggregation.

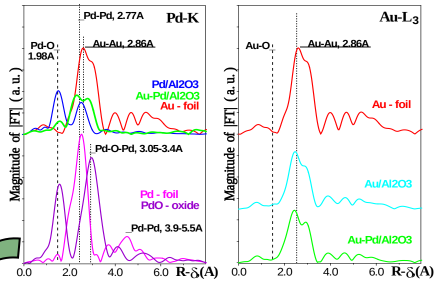
## Main parameters

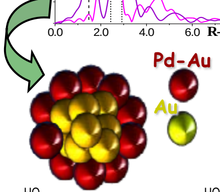
Monochromator: channel cut, silicon, crystallographic planes (111).
The range of photon energies of monochromatic radiation: 5-32 keV
The concentration of the studied element 0.01-100%.
Possibility of measurements techniques - transmittance and fluorescence output (in current and counting modes).

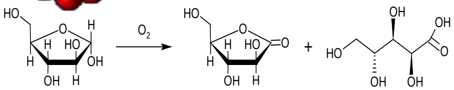
* A study of catalytic nano and precursors for various processes.
* A study of functional nanomaterials, nano-semiconductor, thin nanostructured films.
* Study of organometallic compounds compounds and inorganic complex composition.
* The study of biological objects and archaeological finds.

## X-ray detectors "Scionix" and "Canberra"

## Research applied Pd-Au catalyst raw material processing systems from renewable resources for pharmacology and medicine

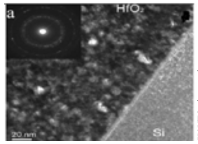



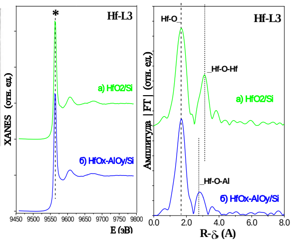




It was found that the structure of the shell of the active component Pd-Au catalysts, leading to high process selectivity.

## Исследование CVD пленок оксидов Hf и Al

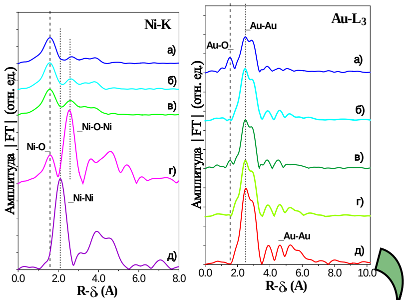


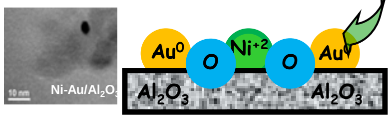
A micrograph of HfO2 / Si cutting and EXAFS data for CVD films based on Al and Hf oxides.




It was established that the method of CVD beta-diketonate complexes Hf and Al nanocrystalline film obtained solid solutions of mixed oxides.

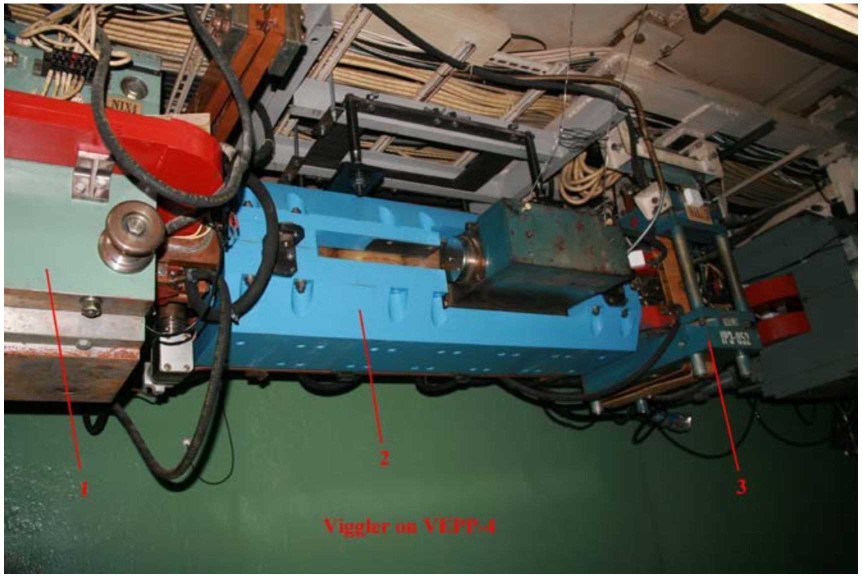
## A study of low-interest Ni-Au catalysts for the conversion of biomass fermentation products for alternative energy

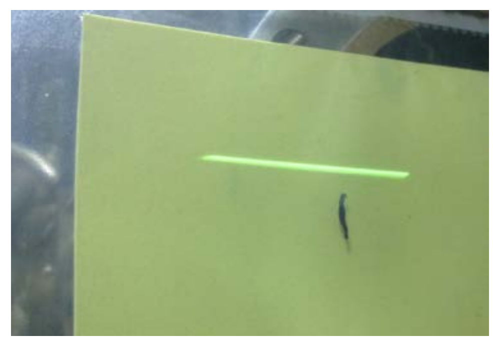




It is shown that the active catalyst Ni-Au component has features of the structure causing high catalytic activity.

# Multipole wiggler on VEPP-4 for hard X-ray applications

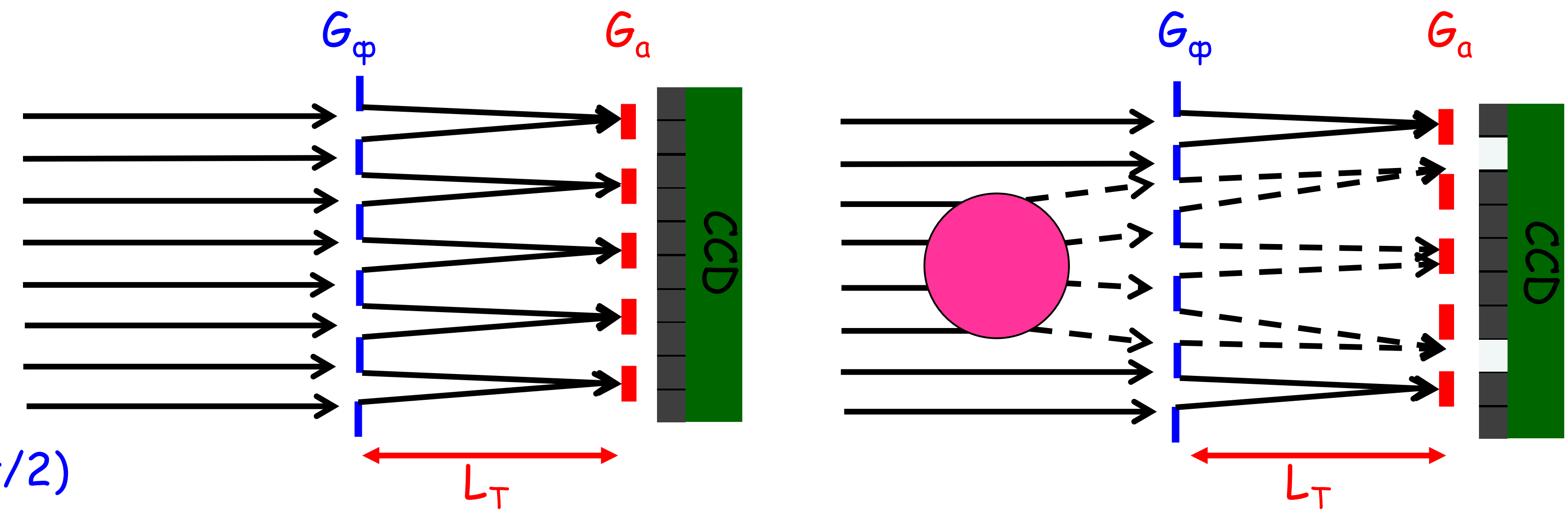



SR beam extraction from 7-pole wiggler VEPP-4M: E = 1.8 GeV (left) and E = 4 GeV (right), I = 40 mA, B = 1.3 T (May 2012).



# New stations on the wiggler beamline from VEPP-4 storage ring

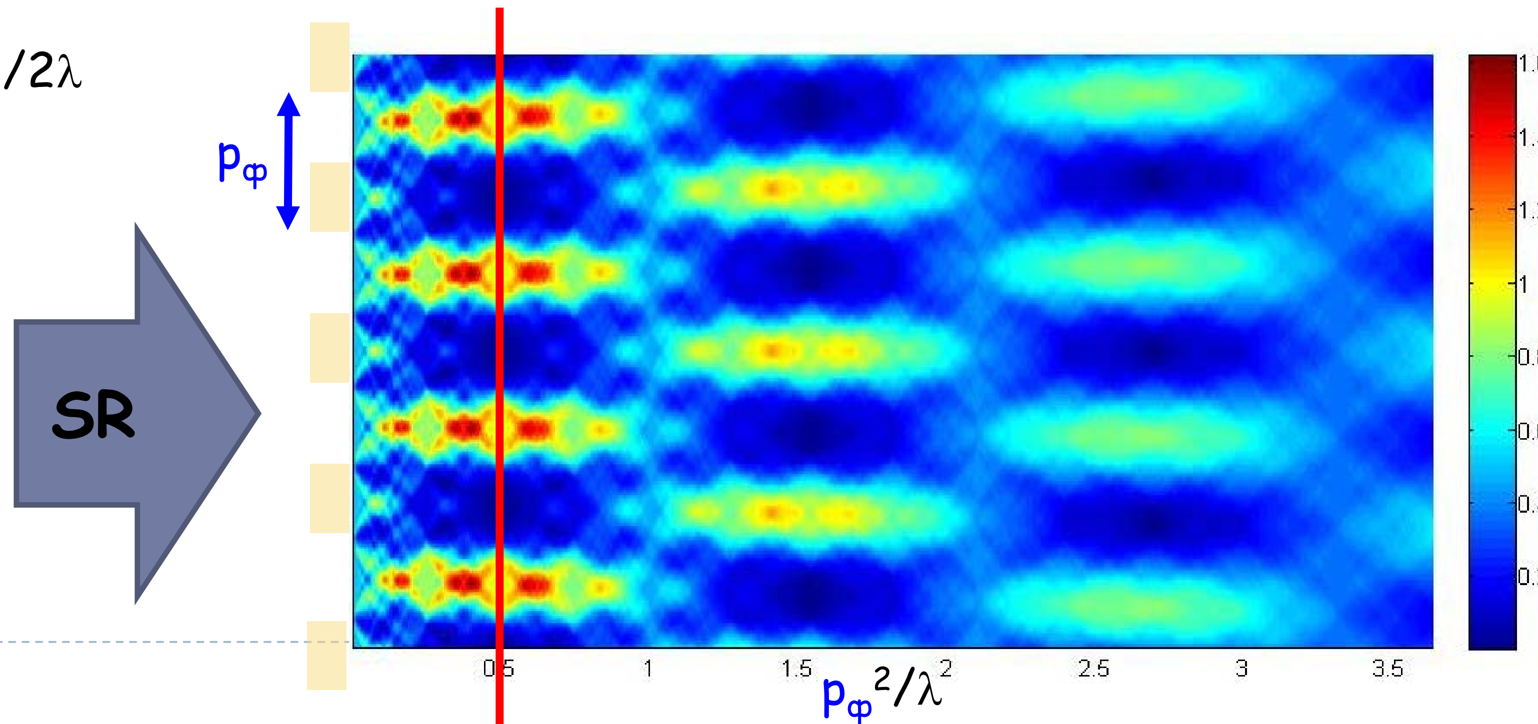
# Phase contrast imaging with using Laue Talbot interferometer



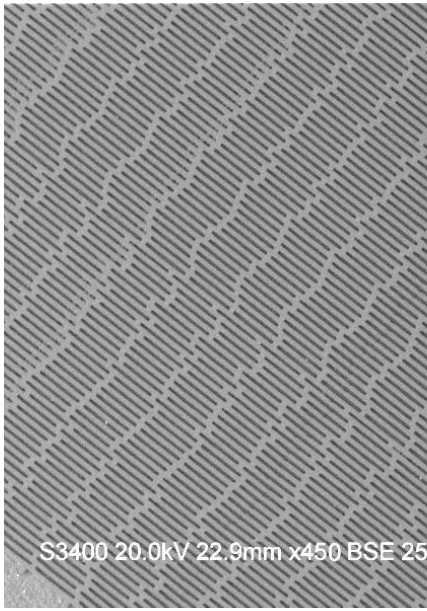

($\Delta\varphi=\pi/2$)

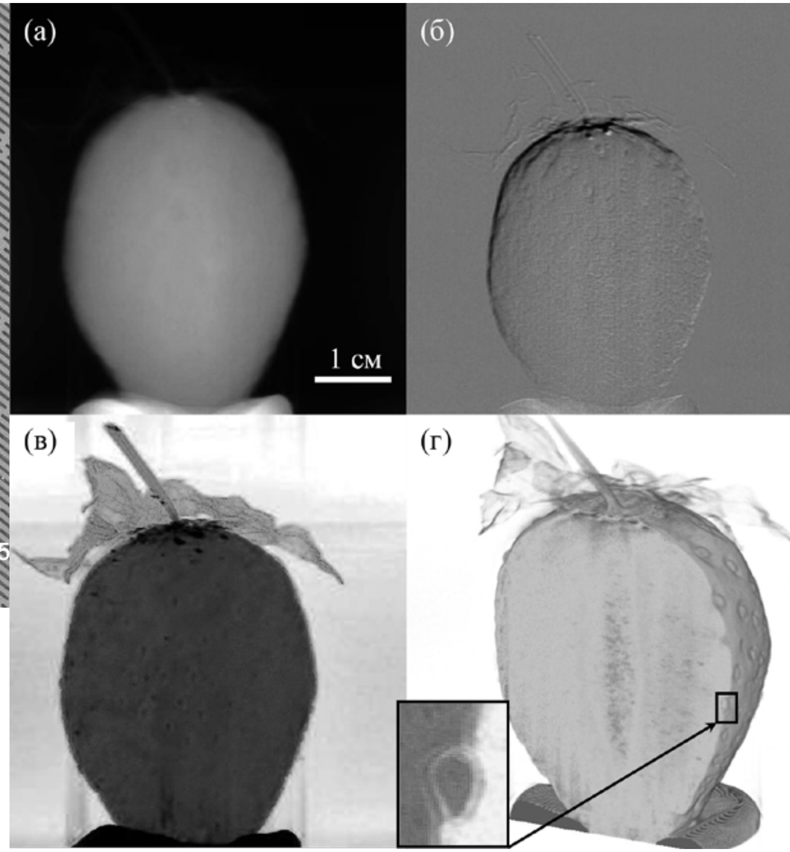
$nL_T = np_\varphi^2/2\lambda$

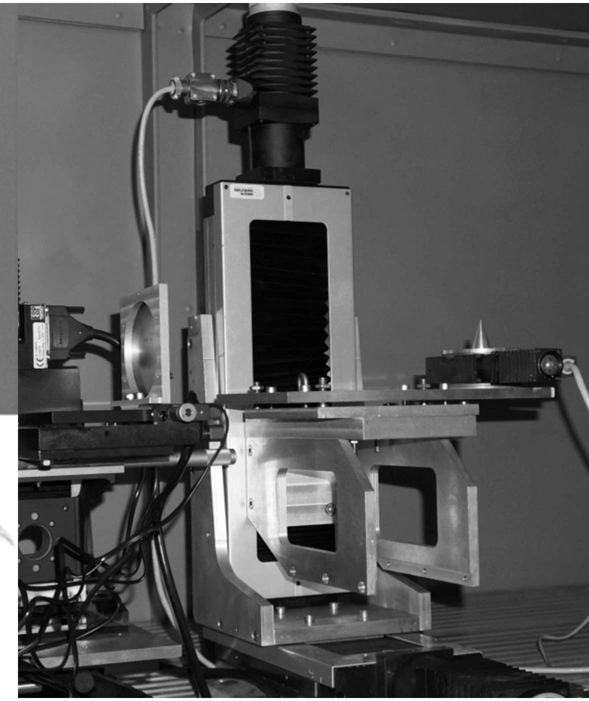
$p_a = p_\varphi$



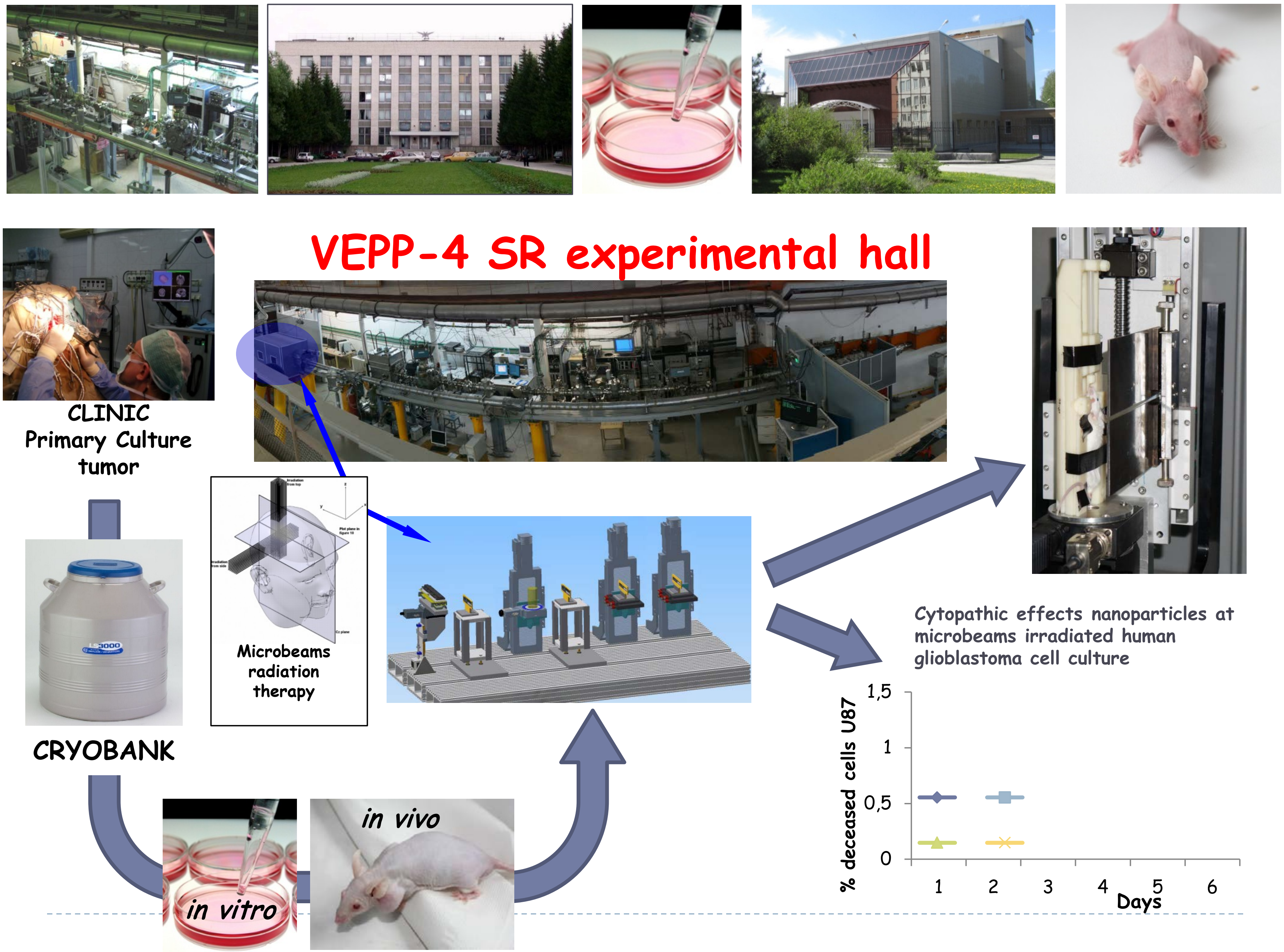
# Phase contrast microscopy



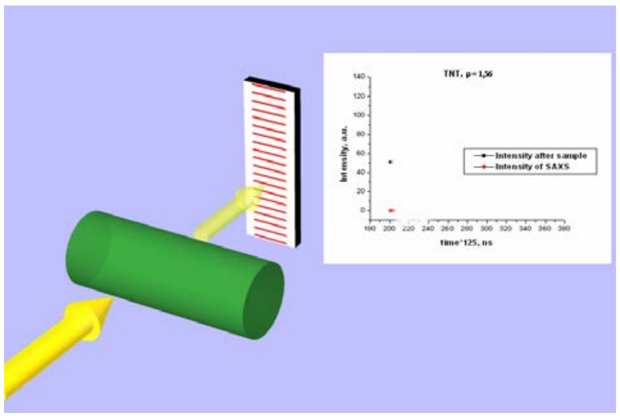



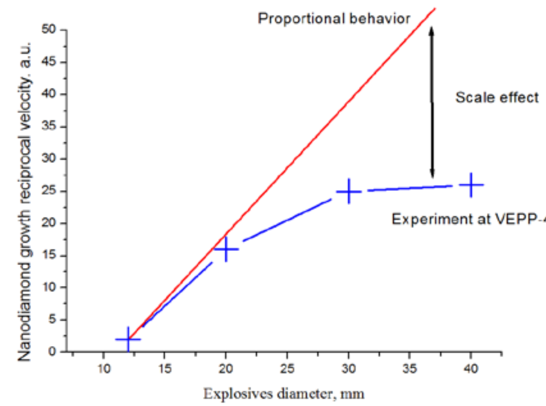
*(a) – Absorption contrast, (б) – Differential phase contrast $\partial\Phi(x)/\partial x$, (в) – phase contrast $\Phi(x)$, (г) – Tomographic reconstruction of three-dimensional structure of strawberries set phase projections.*


VEPP-4 SR experimental hall
CLINIC
Primary Culture
tumor
CRYOBANK
Microbeams
radiation
therapy
in vitro
in vivo
Cytopathic effects nanoparticles at microbeams irradiated human glioblastoma cell culture
% deceased cells U87
1,5
1
0,5
0
1
2
3
4
5
6
Days

# Detonation Diamond nucleation : scale effect



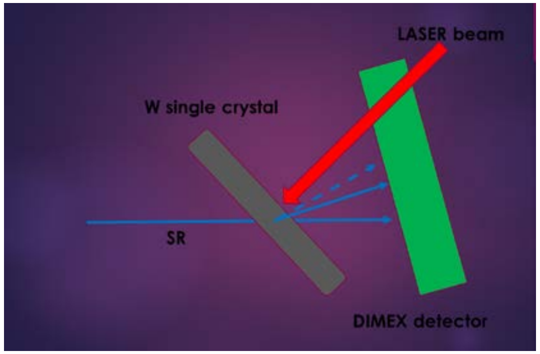
- The scheme of SAXS experiment during detonation of explosive trotyl/hexogen.



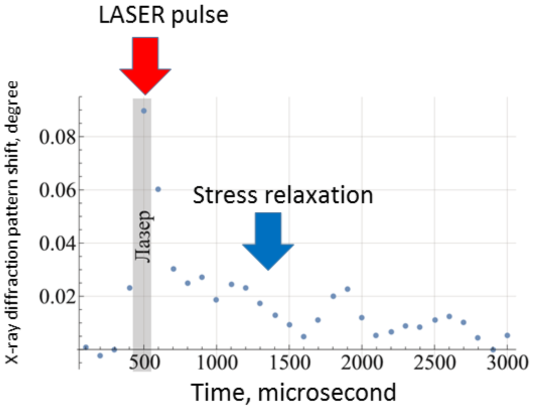

- The scheme of SAXS experiment during detonation of explosive trotyl/hexogen.

- It was found that an increase the mass of explosives leads to increases of produced diamonds mass. Accordingly, increases the rate of formation of diamonds. However, the dependence of the diamonds mass versus the mass of explosive is nonlinear. Also there is non-linear dependence of the formation rate of diamonds versus the weight of the explosives. Thus we observe a scale effect.
- Interpretation: the dependence of chemical reactions from the detonation conditions (diameter), the formation of larger diamonds in the detonation of explosives with large diameters.

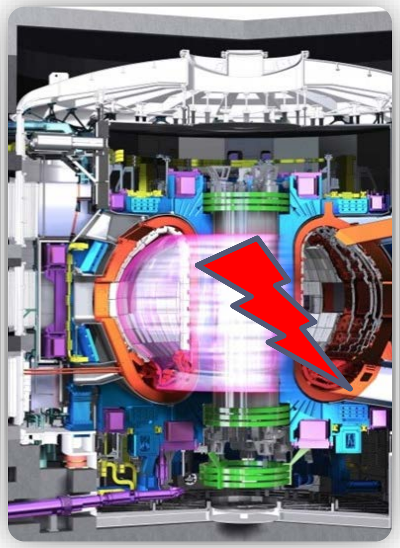
# ITER: plasma discharge on the diverter. Material behavior. Model experiment with laser pulse heating




The scheme of model experiment with LASER pulse heating during 100 microseconds.



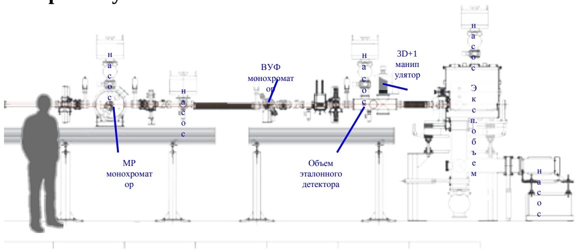

The experimental data of model experiment with LASER pulse heating .



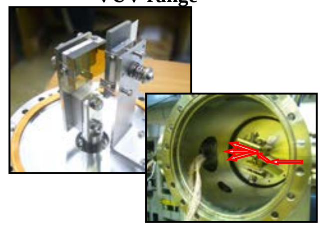
- Now we are preparing an experiment to study the behavior of the crystal lattice of the material of the fusion reactor first wall in a plasma discharge on the diverter

# Soft X-ray and VUV metrology station

## Optics layout




## Gratis monochromator for VUV range



Spectral range: 5 - 100 eV
Spectral resolution: 0.3-2%
The angle of incidence: 70º
Scanning angle: ± 10º
The lattice period: 1/300 mm
Plating: Gold
The fixed position of the output beam in the scanning process - 14 mm

## Soft X-ray monochromator

Spectral range: 80-3000 eV
Spectral resolution: 0.1-10%
The range of angles of incidence: 10º- 85º
Mirrors: Y / Mo, Fe / C, W / Si;
Crystals: mica, RbAP, KAP
Adjust the angle of the second mirror: ± 10º
The fixed position of the output beam in the process of scanning the spectrum

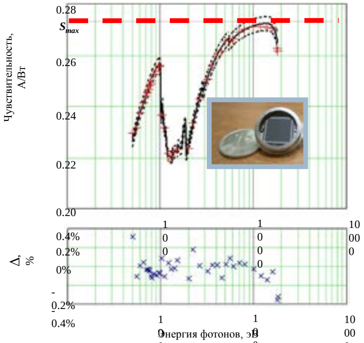
## Reflectometry system in the experimental volume

It allows to work with mirrors, crystals and diffraction gratings. Investigation of the reflection coefficients, rocking curves, quality focusing systems, etc.

The spectral sensitivity of the reference detector SPD
silicon photodiode development
PTI (St. Petersburg)

The calorimeter. Absolute detector for absolute measurement of beam powerof 300 mW or more
Measurement accuracy - 2-5%

The detector is calibrated to the national metrological center German PTB using a cryogenic radiometer. Calibration accuracy - 1%.

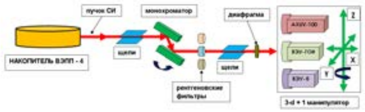


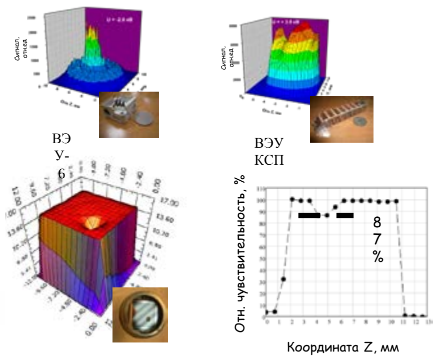

The top graph - the calibration data (+) and approximation of data (solid line) of the model function. The lower graph shows the difference between (x).

## Two coordinate detector fro Lebedev Institute (Moscow)

*Based on CCD E2V tech. (GB)*

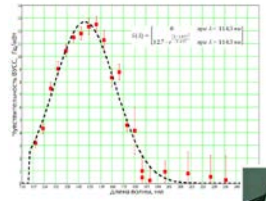
## Sensitivity map measurements




*Map sensitivity photodiode FDUK-100UV after local irradiation dose of 1.8 MGrey (123 J / cm2)*

***Calibration meter solar activity for a geostationary satellite "Electro-L №3" Customer - Institute of Applied Physics (Moscow)***



*Absolute spectral sencitivity*


*certified measurement procedure*

# New light source for SSRC

## Problems

- Currently used storage rings are not dedicated for SR generation, thus the SR parameters are not satisfying for modern requirements
- VEPP-3 and VEPP-4 are intensively used for high energy physics experiments, thus SR experiments have a low priority in operation time sharing rules

# Reasons for creation new source in Novosibirsk

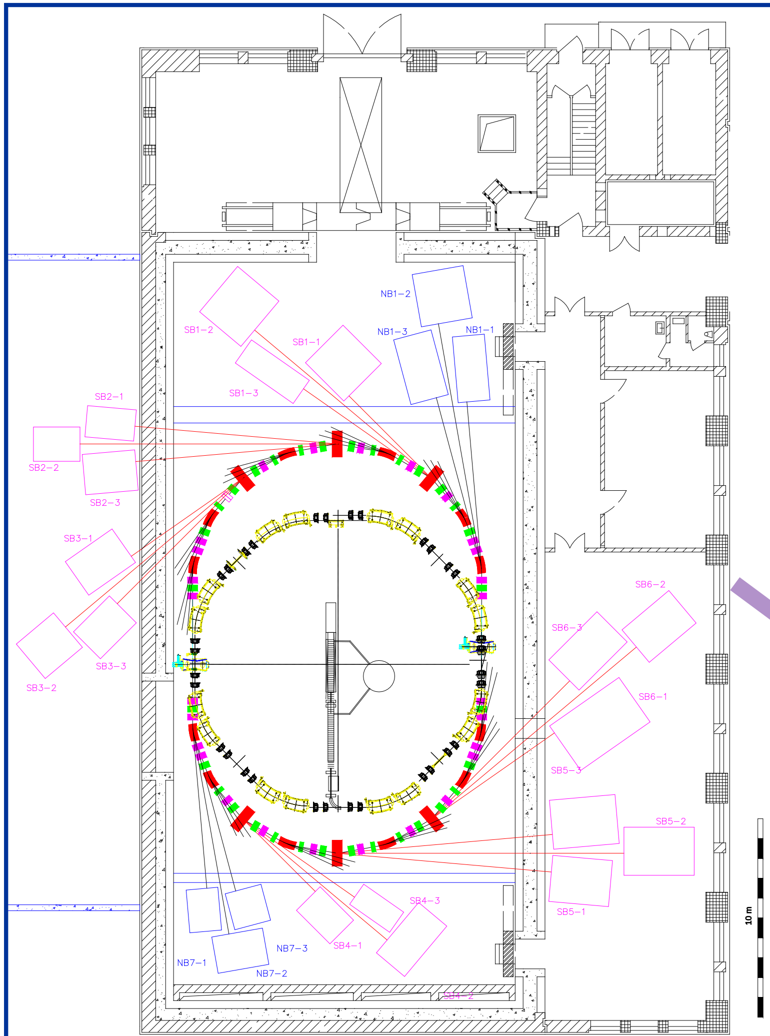
- Siberian Synchrotron Radiation Center (SSRC) unifies many SR users from different scientific organizations. Most popular modern scientific techniques are realized on SSRC beamlines.

- Good geographical location of the Novosibirsk Scientific Center provides effective applications of SR methods for institutes and universities from Siberian region.

- BINP stuff has a big experience for development and fabrication of the modern acceleration elements as well as facilities (including light sources), so possibility to make such source for own needs is evident.

- Great experience of BINP in developing and fabrication of superconducting insertion devices for SR centers also gives some additional kicks for SR source project.

# Proposal history Compact light source with superconducting dipoles




**Основные параметры**

П=55.8 m

$Q_x/Q_z$ = 4.10/3.67

$C_x/C_z$ = -26.78/-9.64

$J_x$=1.038

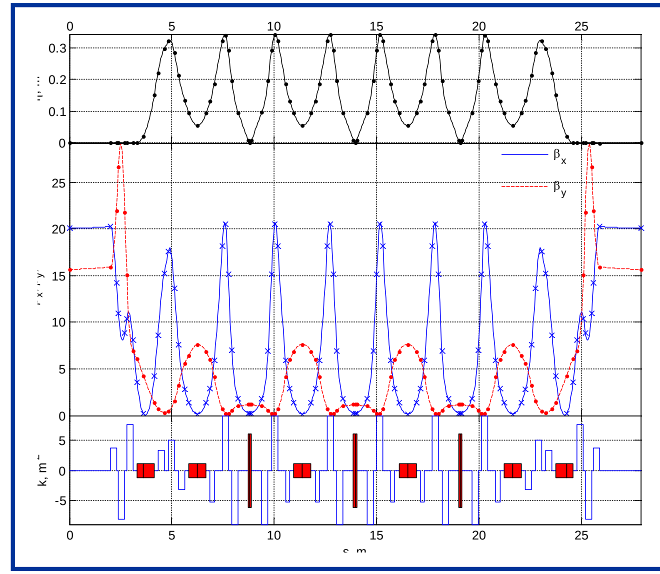
α=4.74 $10^{-3}$

δE/E=1.33 $10^{-3}$

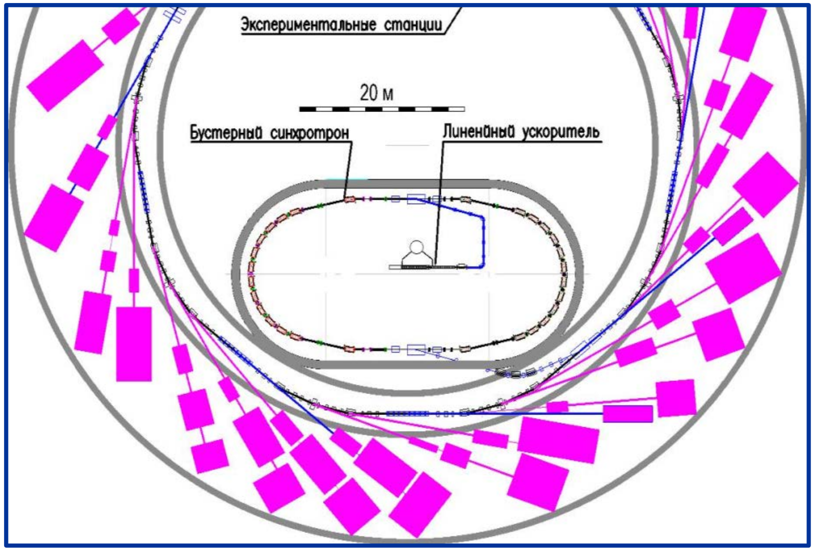
$\varepsilon_x$ = 10.2 nm rad

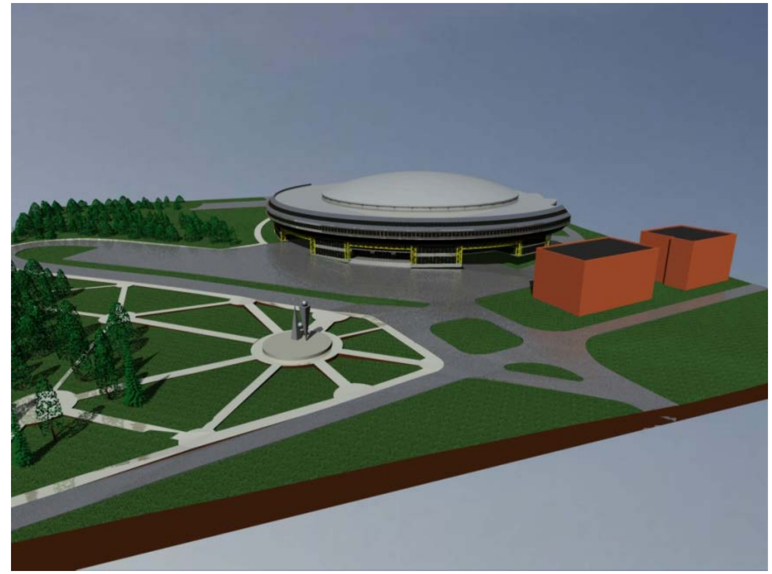
ΔE = 180.2 keV/turn

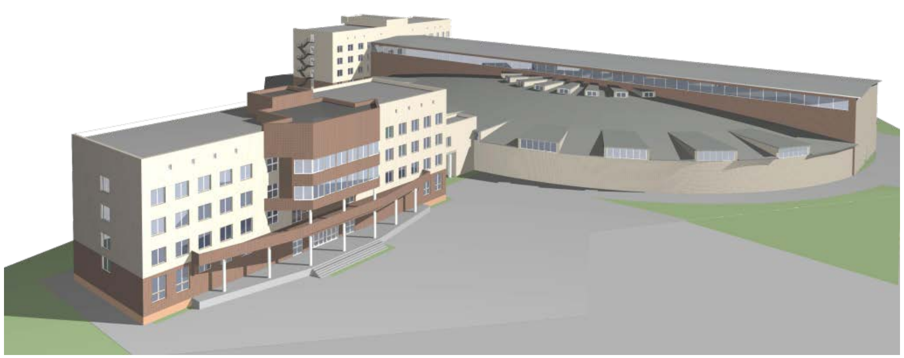
τ = 2.38 ms

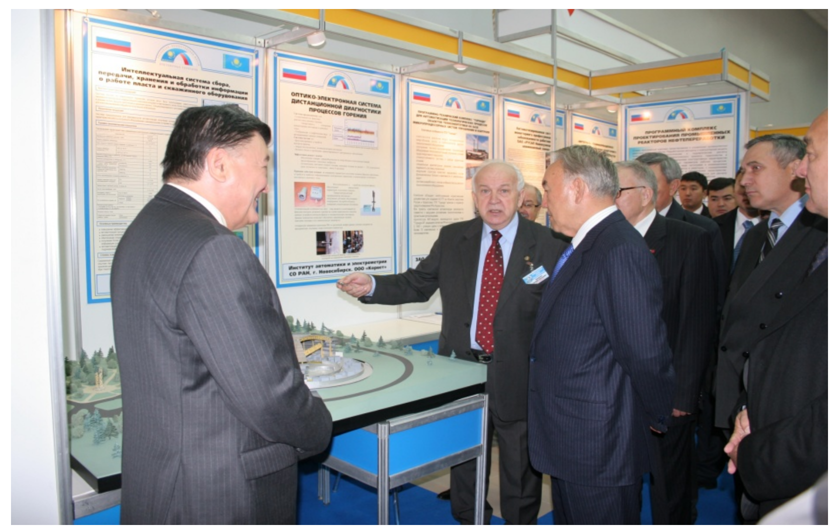



# Proposal history
# 2.2 GeV light source

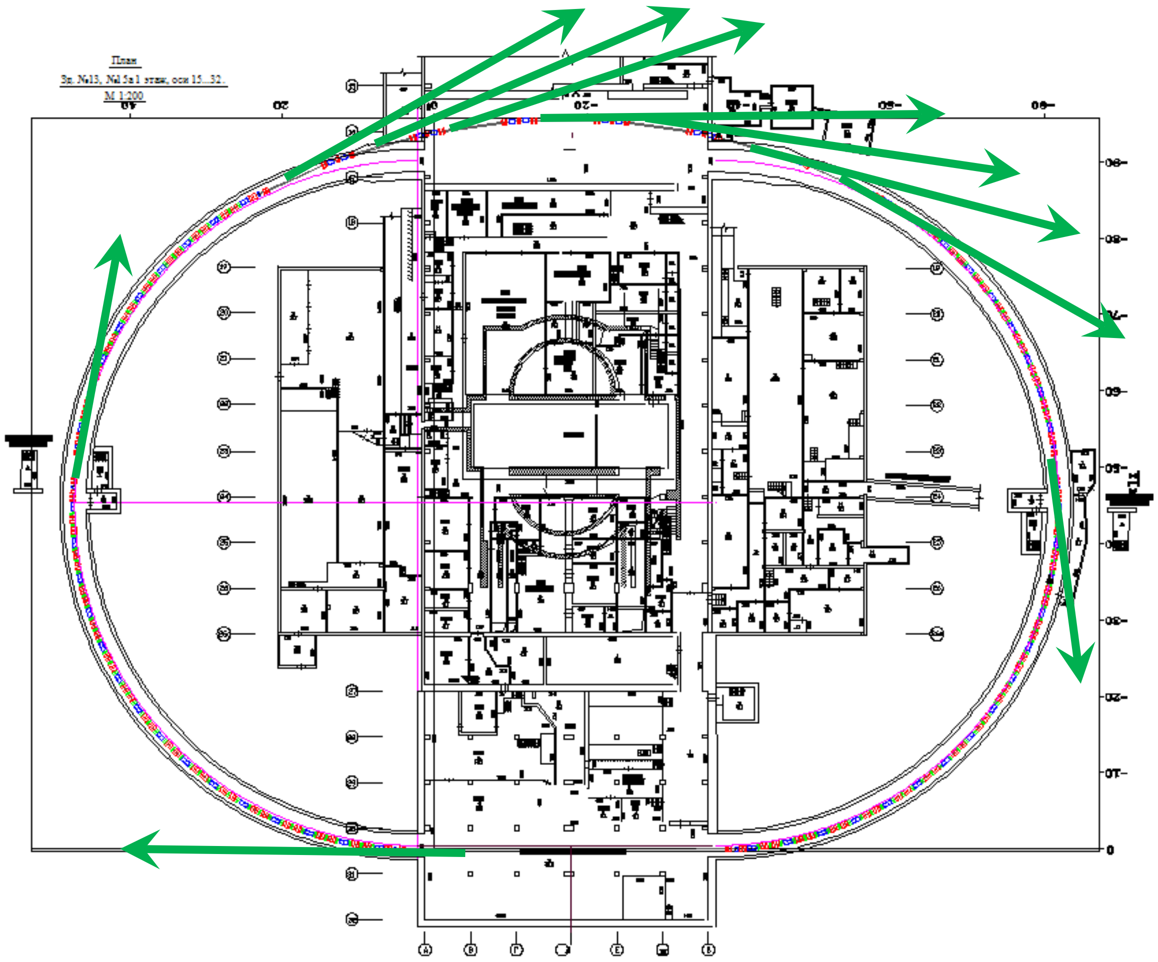




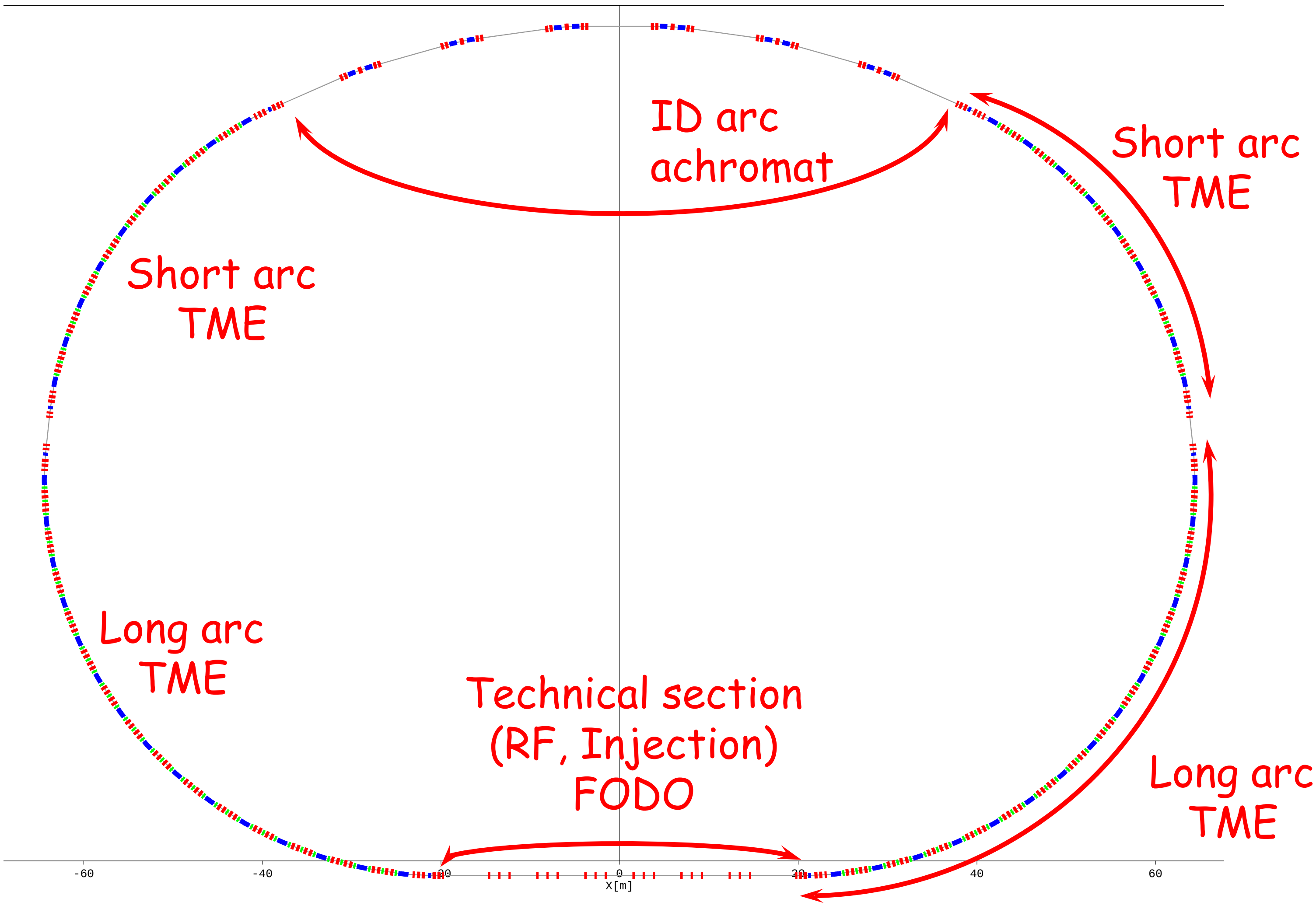


# New light source in the VEPP-4 tunnel

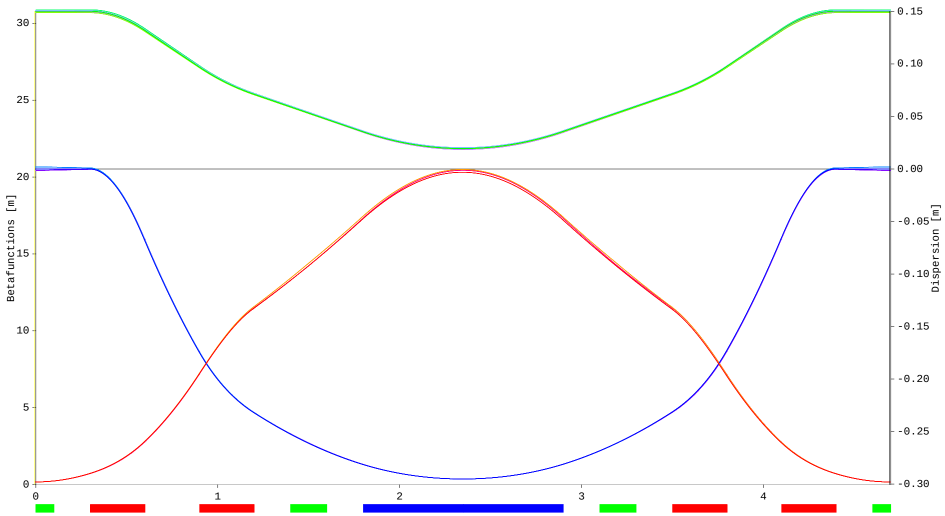


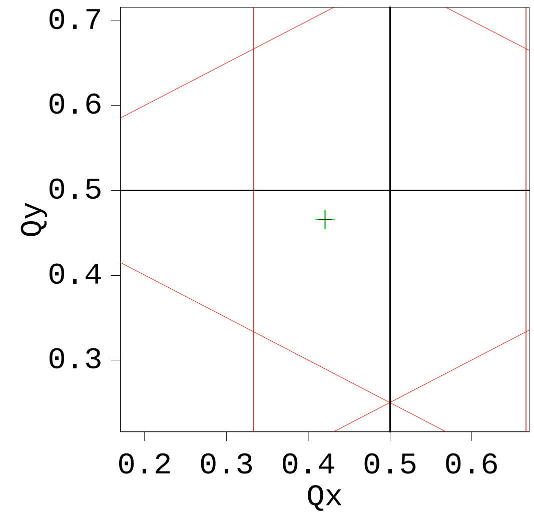
| | |
|---|---|
| Energy | 3 GeV |
| Beam current | Up to 500 mA<br>(2.5 mA/bunch) |
| Emittance | ~ 1 nm rad |
| Injection | 500 MeV |
| Circumference | ~ 360 m |
| IDs | 10 Wigglers and undulators |
| RF | 180 MHz, currenly used |

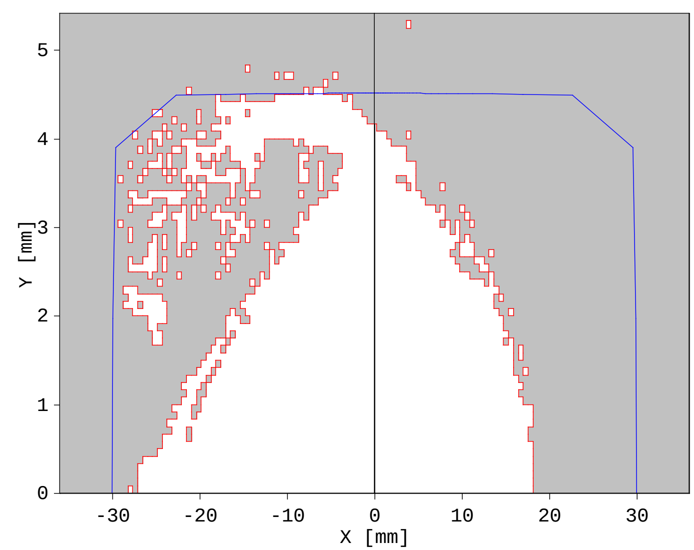
# Ring structure



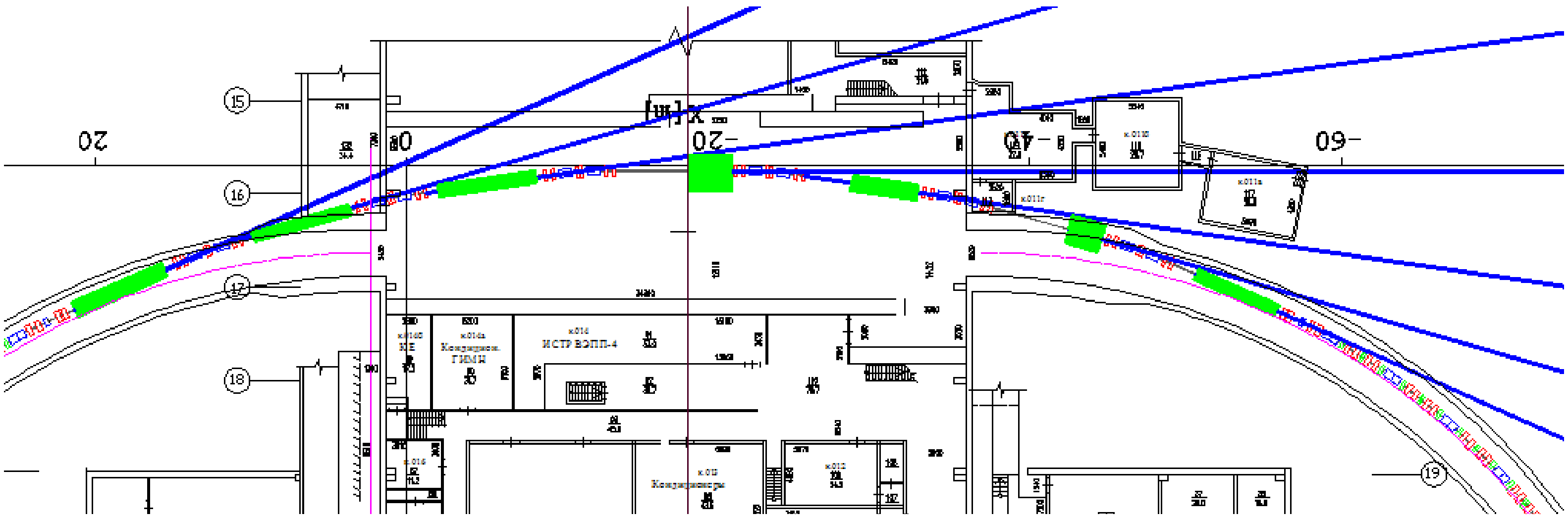
# TME cell

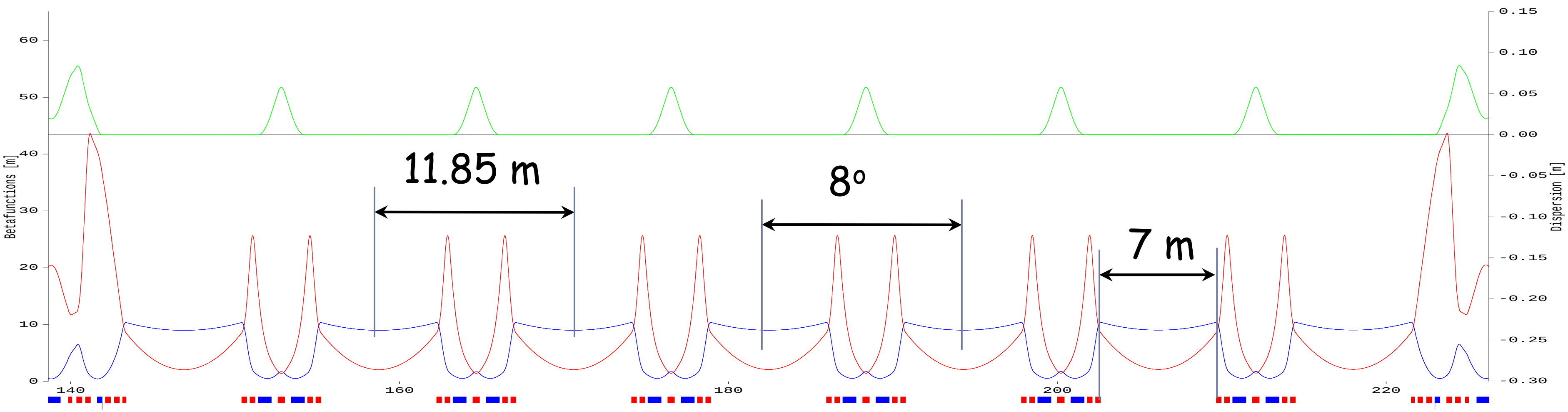

Betafunctions [m]
Dispersion [m]
0 5 10 15 20 25 30
0.15 0.10 0.05 0.00 -0.05 -0.10 -0.15 -0.20 -0.25 -0.30
0 1 2 3 4


Qy
0.7 0.6 0.5 0.4 0.3
Qx
0.2 0.3 0.4 0.5 0.6


Y [mm]
0 1 2 3 4 5
X [mm]
-30 -20 -10 0 10 20 30

# ID section




11.85 m
8°
7 m
Betafunctions [m]
Dispersion [m]
0
10
20
30
40
50
60
140
160
180
200
220
0.15
0.10
0.05
0.00
-0.05
-0.10
-0.15
-0.20
-0.25
-0.30

# Cost estimation

| Component | Price, k$ | Quantity | Total cost, M$ |
|---|---|---|---|
| Dipoles | 30 | 70 | **2.1** |
| Quadrupoles and sextupoles | 15 | 400 | **6** |
| Power suppliers | 1000 | 1 | **1** |
| Diagnostic | 1000 | 1 | **1** |
| Vacuum chamber, 1 m | 2 | 360 | **0.72** |
| Vacuum system (pumps and power suppliers) | 1000 | 1 | **1** |
| Transport channels | 2000 | 1 | **2** |
| Injection system (kickers, septum) | 500 | 1 | **0.5** |
| Insertion devices | 1000 | 7 | **7** |
| Beamline (frontends, shutters, hatches) | 2000 | 10 | **20** |
| Civil engineering works | 2000 | 1 | **2** |
| Total | | | **43.32** |

# Thank you for attention