Lectures - Tuesday, June 17, morning

L5

DESIGN OF INFRARED AND SOFT X–RAY BEAMLINES

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The synchrotron beamlines are complex devices that represent the state of the art in many instrumentation branches, namely in optics, optical technology, mechatronics, precision mechanics, vacuum physics as well as surveying techniques. Although the development of this instrumentation is very fast and successful, there are still problems to be solved.

The beamline is a scientific instrument that transfers, collimates, monochromatizes and focuses a beam of elementary particles, in our case photons, into an experimental station. The beam in the experimental station excites (usually) a secondary radiation that is then collected and analyzed.

The synchrotron radiation beamlines belong nowadays to standard scientific instruments even though each beamline is unique. A big variety of beamline designs have been developed within about half a century. However, there are common issues that must be addressed in any case: optics together with the source choice, vacuum, mechatronics and thermal problems.

The source of light can be characterized with source size $_x$, $_y$, source divergence $_x$', $_y$', photon energy E distribution and photon flux *F*. One uses the derived parameters as emittance (or éténdue), brilliance and brightness. The emittance should not increase during the passage in the optical system, brilliance and brightness should not decrease in the beamline [1].

The most common source of synchrotron radiation is a bending magnet. The bending magnet is an inherent component of a synchrotron storage ring. It emits radiation with a continuous spectrum from infrared to hard X–rays. The bending magnet radiation is linearly polarized in the orbit plane and elliptically above and below it. The bending magnet has its virtues and usage for lithographic, infrared and broad band photoemission beamlines.

Another type of the source is a wiggler. The wiggler is an insertion magnetic device that influences the electron trajectory in a straight section of the synchrotron storage "ring". A periodical array of magnets deflects laterally deflect the electron beam trajectory producing broad band photon spectrum with higher intensity than a bending magnet.

An undulator is an insertion device that differs from the wiggler in a number of magnetic poles and their magnetic field, namely with a bigger number of periods (in the range of 100) and smaller magnetic field than a wiggler. The qualitative change in the radiation is caused by interference of light; the electron beam, that "generates" radiation, is slower than the photon beam that travels with the speed of light.

The current development of undulators goes in the direction of undulators producing variably polarized light. One of the most common examples is the elliptically polarizing undulator Apple II [2].

Almost all the optics used for broad band radiation is reflective. Thanks to rapid technology development, one can use aspherical shapes besides plane and spherical ones. The figure of merit of the optical surfaces for the synchrotron radiation is the parameter slope error. The smallest attainable slope errors are about 0.1 microradians for plane and spherical elements, and about 5 microradians for the aspherical ones.

Surface roughness of the optical surface is another important factor that influences its specular reflectivity. Surface roughness down to 0.5 nm (rms) is now standard achievable.

The most common materials for the synchrotron radiation optics are silicon, GlidCop, Zerodur (glass) and fused silica.

The specular reflectivity of the optical elements for photon energies higher than 5 eV is poor and depends on incidence angle as well as on the surface layer material [3]. Usually, a so–called "grazing" incidence angle (from 89° to 80° to the normal) is often used.

At grazing incidence, effects of the meridional (along the beam on the optical surface in the plane of incidence) and sagittal (perpendicular to the beam on the optical surface) slope error can differ very much. It is desirable to use sagittal focusing, since the impact of the slope error is in this case diminished by a "forgiveness factor".

Gratings represent the most crucial optical elements for monochromators. The designer should optimize many parameters of the grating in order to match the grating efficiency to the requested tuning range of the monochromator. The properties to be optimized are line density, profile of the grooves and reflective coating.

The vacuum in beamlines is required in order to protect the synchrotron storage ring, the optical elements of the beamline against contamination and the environment in the experimental station. The standard attainable vacuum is ultra high vacuum (UHV) ranging at 10^{-9} to 10^{-10} mbar. Ion getter pumps, titanium sublimation and non–evaporable getter and cryopumps are usually used for at beamlines. Oil–free roughing pumps are nowadays standard. There is still a problem with carbon that is present in the stainless steel vacuum vessels. Carbon diffuses into the UHV and optical elements under the intense synchrotron radiation get contaminated with an unwanted effect of decreasing reflectivity in certain photon energy regions. The aluminum vacuum vessel technology, which is carbon–free, is unfortunately not yet mature enough to be used regularly.

The mechanical design of the optical element manipulators and respective vacuum vessels must provide stability and precision that is better than the quality of the optical elements expressed in the term of slope errors that is in the sub–microradian range. The angular movement of the optical elements is for the beamline of the primary concern; therefore the requirements on angular accuracy, repeatability are much more stringent than these for translations.

The design of the mechanical parts should push eigen–frequencies up using stiff and light parts in the internal mechanics. The girder and supports have to suppress unwanted surrounding exciting oscillations. For this function, a synthetic granite block or hollow steel girders filled with sand or foam are used.

There has been a big development in actuator and encoding technology. 5–phase stepper motors can divide one revolution into 125000 (micro) steps [4], closing the gap between a DC servo and a stepper motor in terms of positioning, but keeping the advantages of the steppers. There are now UHV compatible angular encoders that work in sub–microradian range [5]. We think that the optimum mechanical concept for a precision manipulator in UHV is a suitable 5–phase stepper that actuates through a bellow the mechanics in UHV encoded with an UHV encoder. The encoder gives the "real" angular position of the optical element and thanks to the close–loop operation; it is possible to reach sub–microradian repeatability.

The third generation sources as undulators and wigglers emit high–intense beams. Due to the small specular reflectivity, a portion of radiation is always absorbed in the illuminated optical element. The absorbed energy causes heating of the optical surface. Because of non–zero thermal expansion coefficients of the most materials used for substrates, deformation of the optical surface occurs. The heat should be dissipated through active or passive cooling. The most demanding application use internally cooled or cryogenically cooled silicon substrates. The thermal expansion coefficient of silicon at the temperature of liquid nitrogen is very close to zero.

Finite element analysis is often needed to optimize the cooling schemes and to estimate the magnitude of the thermal induced slope error.

The most common beamline elements are slits, beam defining apertures, mirror units, monochromators and diagnostics units.

The slit opening usually ranges from 0.001 to 1 mm. The design of the slits has to assure that the slit blades open symmetrically and cannot be destroyed by closing to zero opening.

Monochromators represent the heart of soft X–ray beamlines. There are several boundary conditions that influence the design of the synchrotron radiation beamlines:

1. The position of the light source as well as the experimental station is fixed in the space, what concerns the position as well as the direction.

2. The vertical size of the source and opening angle of the radiation in the case of bending magnet and wiggler is much smaller than in the horizontal plane. It is therefore desirable to keep the dispersion plane vertical.

3. The reflectivity of all optical materials in the soft X–ray range is poor. Grazing incidence is required.

The historically first grating monochromator is Rowland monochromator with a spherical grating. The spherical grating disperses and focuses the radiation into the exit slit. The grating curvature radius of the Rowland monochromator is equal to the diameter of the circle where the entrance and exit slit should be placed. This means that the positions of the entrance and exit slits varies with the change of the photon energy. The Rowland monochromator, in its original form is not suitable for the synchrotron radiation application.

This disadvantage of the Rowland monochromator is removed on the cost of adding one more optical element. A plane pre–mirror is inserted in front of the spherical grating. The pre–mirror can change the included angle of the grating and thus keep the input and output arms of the monochromator at constant lengths during scanning of a certain range of photon energy. To cover a bigger span of photon energies, one usually has to exchange a grating in the operation. This type of monochromator is usually called Variable included angle spherical grating monochromator (VASGM) [6].

A plane grating monochromator (PGM) represents a development in the direction of bigger flexibility of a monochromator. One keeps the feature from VASGM, i.e. the plane pre–mirror, and uses the plane grating as a dispersing element. The focusing function of the monochromator is taken over by another added optical element (sphere, toroid, ellipsoid, plane ellipse…) positioned after the plane grating.

The plane grating of a PGM can be then used in a very broad photon energy range (over several octaves). If the beam impinging on the plane grating is not collimated, there are certain functions that determine the incidence and diffraction angles over the photon energy range in order to get correct focusing. However, these angles are not necessarily coincident with the optimum angles for the grating.

This drawback has been overcome with Collimated plane grating monochromators (CPGM) which now represent the most usual monochromator type for undulators [7]. Thanks to the fact that the brilliance of the undulator, i.e. size and divergence of the source, are small enough, one can collimate the radiation with the first mirror, and direct it into a PGM. In this case, one has to fulfill only one condition, namely the grating diffraction equation, which has two unknowns: incidence and diffraction angle. The additional needed condition stems from the grating efficiency map.

The bending magnet emits a broad spectrum of photon energies. The infrared beamlines use the infrared (IR) portion of the synchrotron radiation [8]. The bending magnet emits the IR radiation with a big divergence (tens of miliradians). One has recently discovered that there is another kind of radiation that is created between the edges of the precedent and the respective bending magnet. This (IR) radiation is called edge radiation and is much more intense than the "classical" bending magnet IR radiation.

The IR beamline should collect as much as possible of the IR radiation. This is realized by an extraction mirror that separates the IR and visible light from VUV, soft X-ray and hard X–ray components of the radiation. The most common way is to use a plane mirror that deflects the beam by 90° (usually upwards). The reflectivity for X–rays at a

45° mirror is poor and therefore much of the radiation is then absorbed in the extraction mirror. The absorbed power is for a bending magnet of the third generation storage ring in the range of units of kilowatts. One uses a slotted mirror lets the hard X–ray radiation fan go through or a Beryllium mirror that is partly transparent for the hard X–ray radiation.

The IR beamlines are usually divided into two parts, the first one, close to the storage ring, with UHV environment, and then the second one with HV or poor vacuum conditions. The separation windows, which are transparent for IR radiation, are made from diamond, z–cut quartz or Calcium Fluoride according to the IR range needed.

The instrumentation of the IR and soft X–ray beamlines is developing rapidly with the pushing demands from the experimentalist scientific community and steadily increasing amount of synchrotrons over the world.

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L6

MONOCHROMATIZATION OF THE HARD X-RAY RADIATION

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Nowadays experiments with high energy X-rays often requires probe beams with narrow energy bandwidth. The selection of the finite range of the wavelengths (energies) around given value of wavelength is called monochromatization. Because available synchrotron sources (bending magnets, undulators, wigglers …) of the hard X-ray radiation provides polychromatic beam (white beam) for many application there is a need to select only a small energy band. In the region of hard x-rays mainly absorption filters, X-ray mirrors crystal monochromators and multilayer monochromators are used. Filters and mirrors remove from the incoming spectrum only long or short wavelengths, and therefore are used as premonochromators. Highest energy resolution is achieved by the crystal monochromators. If there is no need for the very narrow wavelength band, multilayer monochromators are often employed. Aim of this lecture is to provide overview about basic principles of the monochromatization of the hard X-ray synchrotron radiation and optics which is utilized for this purpose.

Crystal Monochromators

Crystal monochromators play important role in the monochromatization of the hard X-ray radiation. They usually consist of one or more successively arranged diffractors mainly in Bragg geometry. Crystal monochromators are prepared from perfect crystals such as Si, Ge and for description of their diffractive properties the dynamical theory of the X-ray radiation is used [1]. Spectral and angular properties of the one or more successfully arranged monochromators can be visualized with very useful graphic tool called DuMond diagrams [2]. Using these diagrams one can estimate width of the spectral range passed by the monochromator as well as the input angular acceptance and the output angular divergence. Figure 1 is showing the DuMond diagram for a single crystal mono -chromator.

For monochromatization of hard X-rays multiple crystal arrangements are utilized rather than single crystal monochromators (Figure 2). The basic multiple crystal arrangements (Figure 3) consists of the non-dispersive (+, -) and the dispersive (+, +) configuration which properties are analyzed into details. The attention is paid to the double crystal monochromator (DCM) in non-dispersive (+, -) arrangement, which becomes a standard for hard X-ray monochromatization at synchrotrons. Possibilities how to reject harmonics, to increase the resolution by detuning and more others are explained. By utilizing asymmetry angles one can increase the acceptance or increase the angular resolution or achieve the compression or the expansion [3, 4, 5] of the beam.

Successive diffractions, dispersive and non-dispersive, are possible to achieve also in the one single crystal. This monochromators are monolithic [6, 7, 8] and have several advantages and also some disadvantages in comparison with polylithic devices. For example the dispersive monolithic configuration is well known as a channel-cut monochromator. This monochromator has naturally adjusted diffractors and the output beam is parallel with the input

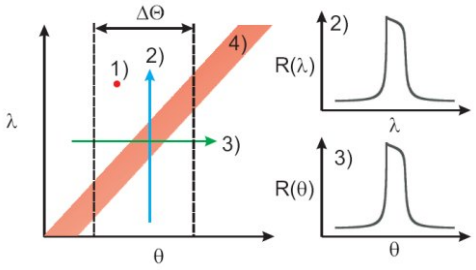

Figure 1. DuMond diagram. Point 1) represents the planar electromagnetic wave, vertical line 2) represents the polychromatic parallel radiation and vertical line represents the divergent monochromatic radiation. The crystal function in this space is represented as a stripe 4).

beam. But by using the channel-cut monochromator we are losing some benefits of the polylithic non-dispersive arrangement. By combining two channel-cut monochromators in the dispersive configuration one can obtain the Bartels monochromator [9]. This monochromator combines properties of dispersive and non-dispersive crystal configurations. Bartels monochromator becomes a standard for laboratory X-ray sources.

For applications where very narrow energy band of the incoming radiation is required in the range of meV and sub meV, it is necessary to accommodate monochromators with higher reflection. There are several possibilities how to achieve sub meV resolution. One of them is to use crystal in so-called backscattering geometry where Bragg angles are close to /2 [10]. Another way is to use dispersive or nested [11] configurations, with higher reflection, where Bragg angles do not need to be necessarily close to /2.

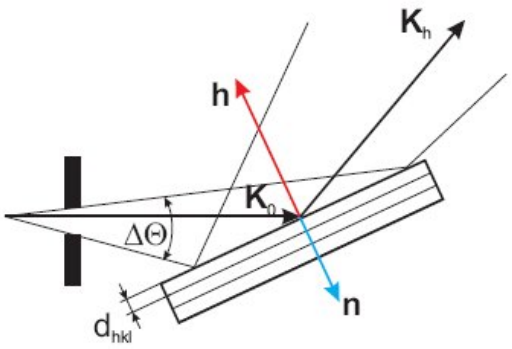

Figure 2. Single crystal monochromator in Bragg configuration.

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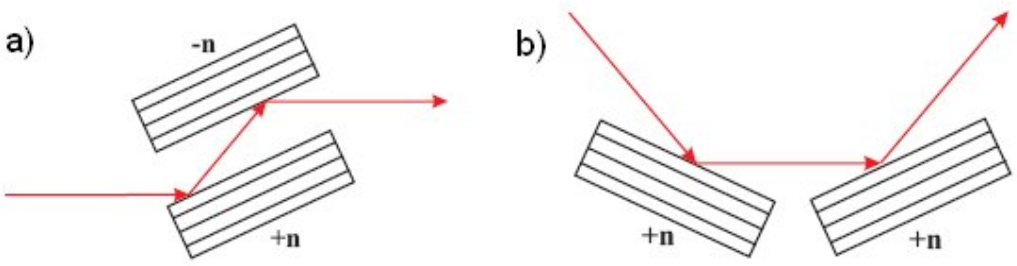

Figure 3. Basic coplanar double crystal monochromators arrangements, a) non-dispersive (+n, -n) arrangement, b) dispersive (+n, +n) arrangement.

L7

NUKLEÁRNÍ REZONANČNÍ ROZPTYL A MÖSSBAUEROVA SPEKTROSKOPIE S UŽITÍM SYNCHROTRONOVÉHO ZÁŘENÍ

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Jaderný rezonanční rozptyl (nuclear resonance scattering, NRS) je proces, kdy jádro s rozdílem energií E_0 základního a excitovaného stavu interaguje s elektromagnetickým záření o energií E_0 nebo jen o málo se lišící. Tato interakce může probíhat buď za účasti nebo neúčasti fononů. Podle toho, zda se jádro před rozptylem nachází ve stejném stavu jako po něm a zda rozptýlené záření je koherentní či nikoliv, můžeme rezonanční rozptyl rozdělit na koherentní elastický, nekoherentní elastický, koherentní neelastický a nekoherentní neelastický rozptyl. Zde se budeme podrobněji zabývat dvěma v experimentech nejčastěji využívanými procesy a to elastickým koherentním rozptylem a nekoherentním neelastickým rozptylem.

Synchrotronová Mössbauerova spektroskopie (SMS)

Využití synchrotronového záření jako zdroje pro Mössbauerovu spektroskopii se datuje od roku 1984, kdy E. Gerdau studoval difrakci synchrotronového záření na jádrech ^{57}Fe v yttrito železitém granátu [1]. Již od prvních experimentů bylo zřejmé, že s využitím synchrotonového záření bude nutné přejít od „klasické“ spektroskopie v energetické doméně k měření v časové doméně.

Koherentní elastický rozptyl je proces, kdy je jádrem bezodrazově absorbován foton, který je následně s určitým zpožděním opět bezodrazově vyzářen. Pro soubor jader, bylo ukázáno, že rozptylu jednoho fotonu se neúčastní pouze jedno jádro, ale soubor jader jako celek (např. jádra v krystalu). Mluvíme o takzvané delokalizované excitaci jader. Díky tomu, že jsou jádra excitována jako celek, můžeme pro popis jaderného elastického koherentního rozptylu použít formalismus založený na popisu šíření elektromagnetického záření v optickém prostředí [2], [3].

Excitujeme-li soubor jader ve vzorku krátkým pulzem elektromagnetického záření takové energie, aby došlo k jeho absorpci jaderným systémem, můžeme detektorem umístěným za vzorkem pozorovat vůči excitačnímu pulzu zpožděné záření vznikající postupnou deexcitací jaderného systému. Toto zpoždění je dáno neurčitostí energie excitovaného stavu. Intenzita záření v závislosti na zpoždění za pulzem je Mössbauerovské spektrum v časové doméně. Jádry emitované záření se koherentně skládá a jsou-li jaderné hladiny vlivem hyperjemné interakce štěpeny, jsou v emitovaném záření obsaženy komponenty o různých frekvencích. Interferencí těchto komponent pak dochází ke vzniku charakteristického průběhu intenzity emitovaného záření na čase. V časovém záznamu intenzity vzorkem emitovaného záření se objeví periodické kmity, tzv. „kvantové zázněje“ (quantum beats). V těchto záznějích je právě obsažena informace o charakteru a velikosti hyperjemné interakce. Zde se s využívá pulzního charakteru synchrotronového záření, kdy záření je koncentrováno v krátkých (< 100 ps) pulzech od sebe vzdálených řádově stovky nanosekund. Za každým z těchto pulzů je detekován časový průběh intenzity záření a tyto jednotlivé záznamy jsou pro zvýšení poměru signálu k šumu sčítány do výsledného spektra.

Jak již bylo uvedeno, v záznějích časového spektra je obsažena informace o hyperjemné interakci. Abychom získali její parametry, je nutné provést analýzu experimentálních dat, která se zpravidla provádí přímo v časové doméně. Nejdříve se provede teoretický výpočet spektra, které se později fituje na experimentální data. Pro analýzu spekter se využívá speciálních počítačových programů, které umožňují jak teoretické výpočty, tak fity experimentálních dat. Příklady takových softwarových nástrojů jsou programy CONUSS [4] nebo MOTIF [5] [6].

Analýza je komplikovanější u vzorků o větší tlouš ce, kdy se uplatní mnohonásobný rozptyl a ve spektrech se kromě kvantových záznějů objeví takzvané dynamické zázněje (dynamical beats). Abychom získali skutečné parametry hyperjemné interakce, musí být během analýzy spekter tyto dva jevy od sebe odlišeny.

Jev elastického rezonančního rozptylu je možné pozorovat ve směru dopadajícího záření to znamená v transmisi (nuclear forward scattering, NFS) a díky poměrně velké intenzitě dopadajícího záření také v difrakci, kdy polohy maxim difraktovaného svazku jsou určeny podmínkami Braggovy difrakce (nuclear Bragg scattering, NBS).

Elastický koherentní rozptyl stejně jako Mössbauerova spektroskopie je metodou pro studium hyperjemné interakce v látkách, jsou zde však jisté rozdíly. Jedním z nich je například určování isomerního posuvu, kdy u „klasické“ Möessbauerovy spektroskopie určujeme velikost isomerního posuvu relativně vůči isomernímu posuvu konvenčního zdroje, který zde hraje roli jistého standardu. U SMS takovýto „přirozený“ standart nemáme (nepřidáme-li ho do zkoumaného vzorku uměle), a proto můžeme pouze u spekter s více komponentami s různým isomerním posuvem určit jejich vzájemný rozdíl isomerních posuvů. Jeden z nejdůležitějších aspektů SMS je ten, že díky vysoké intenzitě synchrotronového záření je doba náběru spektra velice krátká, a proto je tato metoda využívána pro studium velice rychlých procesů, chemických reakcí a difúze [7].

Díky tomu, že synchrotronové záření je emitováno v oblasti od desítek eV až po několik desítek keV, je možné pro NRS využít velkého množství jader (^{57}Fe, ^{40}K, ^{73}Ge, ^{119}Sn, ^{183}W, ^{149}Sm, ...), což dává potenciální možnost studovat hyperjemnou interakci v řadě materiálů, u nichž ještě zkoumána nebyla.

Neelastický nekoherentní rozptyl

Druhou významnou metodou využívající nukleárního rezonančního rozptylu je neelastický nekoherentní rozptyl (nuclear resonant inelastic X-ray scattering, NRIXS). Jestliže se energie dopadajícího fotonu jen o málo liší od energie jaderného přechodu, může dojít k absorpci fotonu spojené s emisí nebo absorpcí fononu a následné emisi fotonu nebo konverzního elektronu. V obou případech je emise vůči absorbovanému fotonu zpožděna, kdy zpoždění je dáno dobou života excitovaného stavu. Ozařujeme-li vzorek krátkými pulzy synchrotronového záření, můžeme snadno od sebe separovat příspěvek pocházející z rezonančního rozptylu na jádrech od příspěvku ostatních rozptylových procesů a to tak, že emitované záření detekujeme až několik desítek ns za pulzem synchrotronového záření, například v časovém okně 30 600 ns za pulzem. Budeme-li postupně měnit energii dopadajícího záření a detekovat integrální intenzitu rezonančně rozptýleného záření, získáme hustotu pravděpodobnosti rezonančního rozptylu v závislosti na energii. V této závislosti je obsažen příspěvek od elastického i neelastického rozptylu. Pravděpodobnost neelastického rezonančního rozptylu je úměrná hustotě stavů fononů, a tedy odejmutím příspěvku elastického rozptylu získáme hustotu stavů fononů (fononové spektrum).

Podobně jako u SMS se ani u této metody neobejdeme bez podrobného teoretického rozboru dějů, ke kterým v látce dochází při neelastickém rozptylu . Tento popis vypracoval V. G. Kohn [8] a Singwi [9]. Pro odejmutí příspěvku elastického rozptylu bylo vyvinuto několik softwarových nástrojů,např. program PHOENIX [10].

Pro tuto metodu jsou klíčové parametry monochromátoru, nebo rozlišení této metody je dáno šířkou pásma monochromatizovahého záření. V současné době již existují monochromátory s energetickým rozlišením pod 1meV, což je dostatečná přesnost pro experimenty NRIXS. Výhodou této metody pro studium fononů je především to, že na rozdíl od měření pomocí pomalých neutronů, kde hustotu stavů získáváme z měření disperzních relací, zde získáváme hustotu stavů fononů přímo.

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L8

INVESTIGATION OF NANOSTRUCTURES BY SYNCHROTRON RADIATION

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Physical properties of nanostructures (quantum wires and dots, nano-rods, metallic nanostructures, among others) are substantially influenced by their morphology, lattice structure and lattice perfection. For the investigation of these structure parameters, X-ray small-angle scattering and X-ray diffraction are the methods of choice. Since the useful scattered signal is usually proportional to the volume or square of the volume of the objects, in the case of nanometer-sized objects the application of synchrotron radiation is inevitable.

In addition to a very high flux and brilliance of a synchrotron beam, which makes it possible to perform "standard" X-ray experiment on very small objects, other unique properties of synchrotron radiation are used for the study of nanostructures, such as a very high beam coherence, ideal polarization of the beam, and tunable photon energy. These properties enable us to measure X-ray scattering from a single nano-object, to carry out absorption spectroscopy studies in one nano-object (methods EXAFS, XANES and DAFS) and to determine the phase of the scattered radiation.

The talk will summarize basic experimental and theoretical approaches for synchrotron studies of nanostructures and will present several experimental examples, concerning mainly semiconductor quantum dots, quantum wires and nano-rods.