Institute of Physics –
Center for Solid State Physics and New Materials
Belgrade, May 2009
Serbia

Center of Excellence in Optical Spectroscopy Applications in Physics, Material Science and Environmental Protection

Center for Solid State Physics and New Materials

Foreword

The Center for Solid State Physics and New Materials (CSSPNM) is a part of the ***Institute of Physics*** *at the University of Belgrade. Our research interest is mainly concerned with the study of optical, transport and magnetic properties of a wide group of materials (from semiconductors and high-temperature superconductors to insulators and magnetic materials). The principal interests of the Center are the vibrational properties of these materials. The present experimental methods include the Brillouin and Raman scattering, photoluminescence, ellipsometry, optical reflectivity and transmission measurements (from far-infrared to UV spectral range), AFM and STM measurements, as well as other optical measurements in a wide spectral range under high pressure and low temperatures. In addition, magnetic and transport properties are investigated using the 14 tesla cryogenic free magnetic system and the Hall-effect set-up, respectively. There is also a substantial theoretical effort in computing the phonon and magnon dispersions of the materials under investigation.*

The CSSPNM also uses different techniques for the synthesis of samples including sintering methods, sol-gel technology, single crystal growth techniques (Bridgman, Czochralski, floating zone, etc.), thin-film technology (thermal evaporation and spattering) including photolithography and impurity doping.

The topics in the focus of recent activities are the theoretical and experimental studies and numerical simulations of various properties of nanostructured systems like high-k wide band gap semiconductors and insulators (such as CeO_2, TiO_2, ZnO), nanosized ferroics as $BiFeO_3$ doped with different rare earth elements such as Nd, Gd, Sm and Y. The vibrational, magnetic and electronic properties of strongly correlated electron systems (vanadates, titanates, manganates), the light scattering by spin waves in oxides, impurity effects in semiconductors and high T_c superconductors, to mention just a few of them, are subject of our permanent interest. Properties of photonic and meta-materials are also some of the main investigation directions in our research.

In this booklet we will shortly present our mission and strategy, current projects, as well as our resources.

Belgrade, May 2009.

Prof. Dr Zoran V. Popović,
Director of Center for solid state physics and new materials

Content

Short history

The Institute of Physics was co-founded in 1961 by the University of Belgrade and the Government of Serbia. The Institute of Physics was established with the following principal objectives in mind:

1. Investigations in all research field in physics, particularly:
   - Research in solid state physics.
   - Research of gas discharge processes.
   - Nuclear physics research.
   - Theoretical research, particularly the research into the history and philosophy of physics.
   - Finding a proper way for the application of research results.
2. Taking care of the organization and conduction of postgraduate studies.

Institute of Physics -view from Danube

In 1973 one part of the Institute of Physics became an independent Institute for applied physics. The next transformation of the Institute of Physics was in 1977, when the Institute was divided into four departments, the Department for solid state physics being one of them. In the first 20 years of existence, the research activities of the Institute of Physics were performed at the Faculty of Science and the Faculty of Electrical Engineering in Belgrade.

July 1983 was an important date in the history of the Institute of Physics. It was then decided to move the Institute of Physics to a beautiful new location in Zemun, a quiet part of Belgrade, located on the Danube river bank. Everything else except the relocation was a far from ideal: the building (an old leather factory bombed in the World War II) lacked a proper roof, there were problems getting telephone lines to the site, there were no proper electricity, water and heating facilities, etc. Step by step, the practical problems were solved in the next few years. In 1977 the neighboring building of old brick factory with a huge piece of land became part of the Institute property. This made it possible to conduct extensive expansion and renovation. After a while the Institute library became the largest repository of books and journals from physics in the country. In 1993 a new reorganization of the Institute of Physics introduced Centers instead of Departments. The Laboratory for Solid State Physics became part of the Center for Experimental Physics.

Backyard of the Institute of Physics

In May 1995 the Laboratory for Solid State Physics became the Center for Solid State Physics and New Materials. At the beginning 6 researchers, 10 PhD students and 11 engineers and technicians were employed in the Centre. Three experimental techniques (far –infrared spectrometer, Raman scattering system, Hall-effect set-up) were at our disposal at that time.

The most important day in the history of the Center was June 2006 when the OPSA project, with a budget of €400.000, was signed. The aim of this project was to improve the level of scientific and technological research in the Centre for Solid State Physics and New Materials in order that it should become the European Centre of Excellence for **Op**tical **S**pectroscopy **A**pplications (OPSA) in Physics, Material Science and Environmental Protection. This project allowed for an up-grade of existing experimental set-ups to be conducted, as well as acquisition of new equipment and laboratory space renovation.

Substantial improvements of experimental conditions were carried out in 2007. Thanks to the National Investment Plan of the Republic of Serbia we have acquired some capital equipment, such as a AFM and a STM variable temperature microscopes, a variable angle spectroscopic ellipsometer, a 14 tesla magnetic measurement system, a micro Raman set-up, etc. In the same year we signed two new projects within FP6 and FP7 Programme of the European Community, which further accelerated our development.

Mission and strategy

A general mission of our Center is, according to the constitutional act, to perform research in the field of solid state physics and condensed matter generally. Particularly, to help universities in organizing master and doctoral studies, to collaborate with corresponding scientific and professional institutions all around the world, and to make connections between research and commercial opportunities. Having this in mind we are trying to find a way to increase mutual interaction within the knowledge triangle: education, research and innovation.

Firstly, in order to contribute to the highest level of education, almost all of our researchers are engaged at the Faculty of Physics or the Faculty of electrical engineering, as well as at the Doctoral school of the University of Belgrade, as lecturers. On the other side, a lot of PhD students are doing their doctoral studies in our Center.

Secondly, we promote excellence in basic and applied research as a very important component in the development of our Center. The scientists employed at the Center have published more than **450** scientific papers in well reputed international journals. We organize training courses for the implementation of optical characterization metods in science and industry. Up to now, the Center for Solid state physics and new materials became the European Center of Excellence for Optical Spectroscopy Applications (OPSA) in Physics, Material Science and Environmental Protection and also the National centre of excellence for nanoscience and nanotechnology. We maintain our competitiveness working on European projects in collaboration with many European centres and universities as well as with universities from the USA, Japan and Russia.

Thirdly, to overcome a gap between the research and the application of our knowledge in industry and to support the collaboration with small and medium-size enterprises we have founded two spin-off companies: the Spektroskopija-Infiz and the Kristal-Infiz. The Spektroskopija-Infiz mostly deals with the application of spectroscopic techniques in earth observation research (remote sensing) and applied spectroscopy. The Kristal-Infiz produces various kinds of single crystals for semiconductor and optical industries.

Finally, all our activities are focused on the people. The knowledge everyone is talking about is *their* knowledge, the spirit of entrepreneurship is *their* spirit, the ability and willingness to bring an idea to market are *their* skills.

Research potentials

Optical, transport and magnetic property techniques, such as photoluminescence, ellipsometry, reflectivity and absorption measurements (from far-infrared to far-UV), Raman and Brillouin scattering spectroscopy, Hall-effect measurements, magnetic susceptibility and magnetization measurements, are practical, cost effective methods for the non-destructive characterization of materials, thin films and real device structures. These methods are versatile and can be used on a wide variety of the advanced materials (also on nanosized materials together with AFM and STM techniques) and structures.

The main research activity in the Centre for Solid State Physics and New Materials of Institute of Physics concerns optical, transport and magnetic characterization of a wide group of materials, from metals, superconductors, semiconductors, insulators as well as materials with magnetic properties. In total, 37 people are employed among them 14 researchers, 12 PhD students and 11 engineers and technicians.

The Centre for Solid State Physics and New materials consists of several laboratories:

- *Laboratory for material synthesis and crystal growth,*
- *Laboratory for nanoscopy (STM, AFM, SNOM)*
- *Laboratory for FT-infrared spectroscopy and ellipsometry*
- *Laboratory for Raman scattering and photoluminescence*
- *Laboratory for μ-Raman scattering spectroscopy*
- *Laboratory for Brillouin scattering spectroscopy*
- *Laboratory for transport properties measurements (Hall effect set up)*
- *Laboratory for magnetic and magneto-optic measurements.*

The current projects of the Centre for Solid State Physics and New Materials are:

- **OPSA 026283 SSA Project supported within FP6 Programme of EC[1]**

[1] The aim of this project is to improve the level of scientific and technological research in the Centre for Solid State Physics and New Materials of Institute of Physics in order that it should become the Centre of Excellence for Optical Spectroscopy Applications (OPSA) in Physics, Material Science and Environmental Protection. The OPSA Centre should contribute to improving the existing and establishing new links with European Centers in the field of applications of the advanced spectroscopy techniques in diverse fields of the natural science, through the active exchange of scientists between OPSA Centre and European institutes and universities, international workshops and training of young scientists.
The main objectives of the Centre of excellence for OPSA are as follows:

- To promote long term research into understanding phenomena, mastering processes and developing research tools in the field of Nanoscience and Nanotechnologies through an upgrade and renew of our experimental techniques.
- To develop human potential by educational and training activities.
- To promote cooperative research and technological and educational activities between research centers, universities and industry in the field of Micro- and Nano-technologies and Microsystems.

The OPSA project addresses the following thematic area of FP6 Programme: 3.4.1 *Nanotechnologies and nanosciences*. In particular this project contributes to: 3.4.1.1 *Long-term interdisciplinary research into understanding phenomena, mastering processes and developing research tools.*
Data about project: OPSA 026283 project signed on 29.06.2006; Duration of project is 3 years; Budget: 400.000 Euros
Head of Project: Prof. Dr Zoran V. Popović.

- **CoMePhS STREP Project No. 517039 supported within the FP6 Programme of EC**[2]
- **NanoCharm project no. 218570 supported within the FP7 Programme of EC**[3]
- **NIM_NIL project No. 228637 supported within the FP7 Programme of EC**[4]
- COST Action P16, "ECOM"- 'Emergent behavior in correlated matter."
- COST Action 539 ELENA - "Electroceramics from Nanopowders Produced by Innovative Methods"
- Raman scattering and photoluminescence from semiconductor nanoparticles, Common project with ISSP Bulgarian Academy of Sciences.
- Optical, magnetic, and transport properties of semimagnetic semiconductors, Common project with the Institute of Physics, Polish Academy of Sciences.
- Electronic structure and properties of carbon- and other nano-materials, Common project with Institute of Science of Materials, Ukrain Academy of Science.
- Ionic conductor with perovskite structure in the Sr, Mg- doped $LaGaO_3$ system for SOFC-intermediate temperature, Common project with Institute of Physical Chemistry, Romanian Academy of Science.
- High pressure Raman spectroscopy of low-dimensional magnetic structures, Common project with NTU Athens, Greece
- Interplay of orbital, spin and lattice in strongly correlated electron systems, Common project with ISSP, Tokyo University, Japan
- Spectroscopic and galvano-magnetic properties of manganates, Common project with IMSUV, Valencia, Spain
- Solid State Physics Methods in Study of Medieval Cultural Heritage, Common project with Institute for Applied Physics, University of Florence, Italy
- Physics of low dimensional and nano-sized structures and materials[5]
- Spectroscopy of elementary excitations in semimagnetic semiconductors[6].

For more details and results of our activities see our web site: http://www.solid.phy.bg.ac.rs.

[2] **CoMePhS** 517039 project is FP6 STREP project on Controlling Mesoscopic Phase Separation. It belongs to thematic priority: Nanotechnologies and Nanosciences: Knowledge-based Multifunctional Materials and New Production Processes and Devices. The main objective of the CoMePhS project is precisely to understand, manipulate and control the phase competition to a level that allows mastering of the resulting mesoscopic texture.
Duration of project: 42 months; Budget: 207400 Euros (our share in a 24 month period, EC contribution 50%). Our group leader: Dr Zoran V. Popović. Project ended in November 2008.

[3] **NanoCharm** is a Europen FP7 project on Multifunctional Nanomaterials Characterization Exploiting Ellipsometry and Polarimetry. Objectives of NanoCharm project are:

- to characterize the nanoworld, using ellipsometry and polarimetry
- to provide a platform for nanomaterials and nano applications exploiting ellipsometry
- to develop and refine the nano measurement tools of the future
- to promote education, communication, dissimination of knowledge, networking, research and innovation in characterization of nanomaterials.

Project started on 01. January 2008. Duration of project is 3 years; Budget: 111.000 Euros (our share). Our group leader: Dr Radoš Gajić.

[4] **NIM_NIL** is a Europen FP7 project on Large Area Fabrication of 3D Negative Index Materials by Nanoimprint Lithography, which should start on 01. September 2009. Duration of project is 3 years; Budget: 420.000 Euros (our share). Our group leader: Dr Radoš Gajić

[5] **Project No.141047** is supported by the Ministry of Science and Technological Development of the Republic of Serbia. This project consists of several topic areas: 1. Optical and magnetic properties of nanosized materials and structures, 2. Investigation of strongly correlated electron systems (vanadates, titanates, manganates, etc), 3. Research of photonic and meta-materials. Duration of project is 5 years; Budget: 500.000 Euros/year. Head of project: Dr Zoran V. Popović.

[6] **Project No.141028** is supported by the Ministry of Science and Technological Development of the Republic of Serbia. The optical and transport properties of narrow band gap semiconductor alloys based on the lead and mercury halchogenides, undoped and doped with the elements of the III group or transitional elements with unfilled d-orbital are investigated within this project. Duration of project is 5 years; Budget: 200.000 Euros/year. Head of project: Dr Nebojša Romčević.

Experimental facilities

The Center for Solid State Physics and New Materials consists of several laboratories:

Laboratory for material synthesis and crystal growth consists of several crystal growth techniques such as Czochralski, Bridgman or floating zone, thin film technology methods (thermal evaporation, spattering, laser ablation), sol-gel technology, sintering, etc. Here we will show only two crystal growth techniques.

Crystal growth using floating zone technique: Four Mirror Lamp Image Furnace.

The floating zone technique is a powerful tool for the fabrication of high quality single crystals as well as for the purification of materials. A new four mirror optical floating zone furnace **FZ-T-1000-H-HR-I-VPO-PC** (Crystal System Co.) consists of four ellipsoidal mirrors made of Pyrex glass coated with highly reflective aluminum. The mirrors are air-cooled in order to prevent moisture condensation on the ellipsoidal surfaces. The principle of this technique is that radiation from the Halogen lamps is reflected and focused by the mirrors onto the bar sample to form a molten zone at the tip of the feed rod. Then the molten (floating) zone is translated along the sample length by moving the mirror stage with respect to the sample. The crystal is grown on the solidifying end of the floating zone. In addition, a rotation movement of the rod improves the microstructural homogeneity during directional solidification.

In the crucibleless floating zone technique the molten zone is kept together by capillary forces. The optical heating is the optimal way to bring a narrow zone of the sample to melting. In **FZ-T-1000-**

H-HR-I-VPO-PC optical furnace the maximum operating temperature is 2200°C in atmosphere (air, nitrogen, oxygen, argon, etc) within the pressure range from $5x10^{-5}$ up to 10 bars. Crystals can be obtained up to 150 mm in length and 10 mm in diameter, with a growing rate of 0.05-27 mm/h and a 5-60 rpm rotating rate.

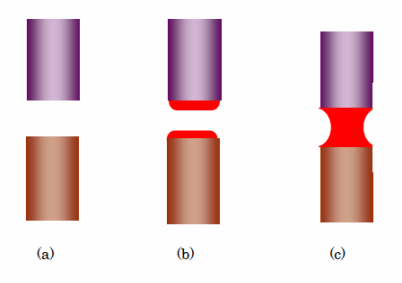
Four mirror optical furnace model FZ-T-1000-H-HR-I-VPO-PC (Crystal System Co.) with vertical molysili furnace model VF1800 (right).

Advantages of the four mirror floating zone furnace:

1) **Stable molten zone -** Using high quality four glass mirrors and aluminum frame, a stable molten zone can be achieved;
2) **Small size -** Mirror stage moving system can diminish the scale of the furnace, so that crystals as long as150 mm can be grown;
3) **Remote monitor and remote control system** - Connect a PC to LAN system, monitoring and controlling of the furnace can be done from anywhere in the world;
4) **PC control -** All the parameters for growing single crystals can be set and controlled by the personal computer;
5) **High quality glass mirror -** Suitable for a long-term use, and optimal conditions are easily maintained. The surface is easy to clean, no damage occurs;

6) **Variety of halogen lamps -** The most suitable lamp can be selected from various power lamps (150W, 300W, 500W, 1,000W and 1,500W lamp are available);
7) **Monitoring by the CCD camera -** High quality color CCD camera and monitor can give real time control of the growth;
8) **Phase research by the slow cooling float zone method -** Stable molten zone by the four mirror system can give the phase relation using the slow cooling float zone method.

Growth of congruently melting compounds

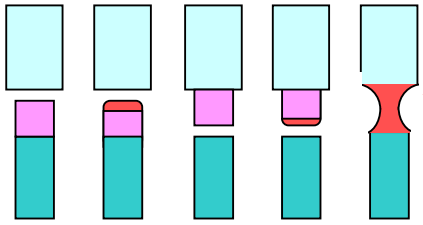


(a) At the initial stage of the heating, both the feed and seed rod are set apart by a distance of a few mm;

(b) As the heating progresses, both ends of the feed and seed rod will begin to melt;

(c) At this stage, the upper feed rod is moved to downwards until it touches the seed rod. Finally, the length of the molten zone is adjusted so that its diameter is almost the same as that of the feed and seed rod

The growth of incongruently melting or peritectic compounds



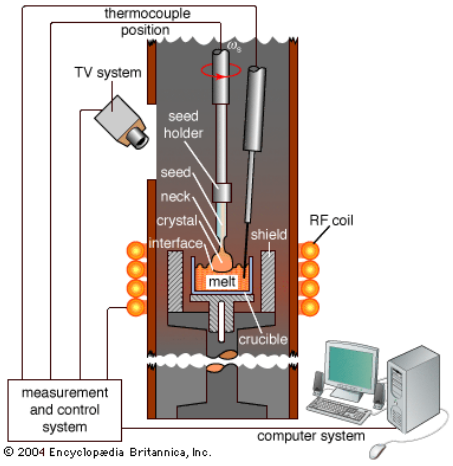
1) **First stage**: Set the solvent zone chip on the top of the seed rod. A small amount of starch paste can be used to fix it. The starch will be burnt off as the temperature increases, and a small amount of smoke will appear.
2) Second stage: Both the top of the solvent zone chip and the end of the feed rod are partially melted, and connected without rotation of either the upper or lower shafts. After connecting, the lamp power is slightly decreased and the upper shaft is retracted to separate the solvent zone.
3) Third stage: Both the end of the solvent zone chip and the top of the seed rod are partially melted, and brought into contact with rotation of both shafts.
4) Final stage: Adjust the lamp power to keep the molten zone stable and to melt a few millimeters of the top portion of the seed. After a few minutes, the crystal growth will begin.

Advantages of Floating zone in comparison with the Czochralski method:

1) High purity crystals can be grown without contamination from the crucible;
2) Can be applied to grow oxide, metal and other materials;
3) Can grow single crystals of incongruently melting materials;
4) Low cost of the crystals growth;
5) Can be applied in phase research by the slow cooling float zone method.

Crystal growth by the Czochralski method

The **Czochralski process** is a method of crystal growth used to obtain single crystals of many semiconductors (e.g. silicon, germanium and gallium arsenide), optical materials (e.g. ruby, sapphire, YAG, $Bi_{12}GeO_{20}$), metals (e.g. palladium, platinum, silver,

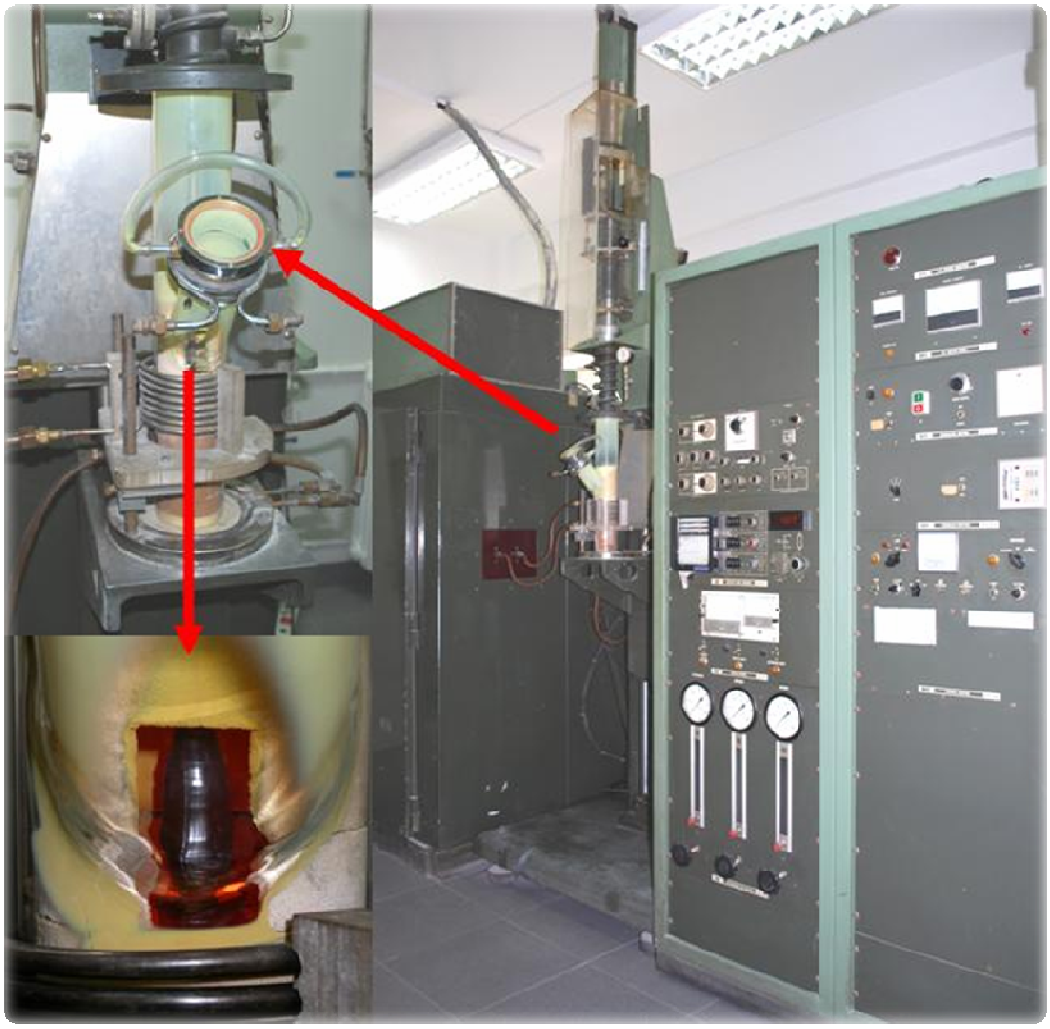


gold), salts and some hand made, (or "lab") gemstones.

High-purity starting materials or polycrystals are melted down in a crucible, which is usually made of quartz, platinum, iridium or molybdenum. A *seed crystal*, mounted on a rod, is dipped into the melt. The seed crystal rod is pulled upwards and rotated at the same time. The dopant impurity atoms can be added to the melt. By precisely controlling the temperature gradients, the rate of pulling and speed of rotation, it is possible to extract a large single-crystal cylindrical ingot from the melt. This process is normally performed in an inert atmosphere, such as argon, or in an inert **chamber, such as quartz.**

Occurrence of unwanted instabilities in the melt can be avoided by investigating and visualizing the temperature and velocity fields during the crystal growth process.

The process is named after Polish scientist Jan Czochralski, who discovered the method in 1916, while investigating the crystallization rates of metals.



Czochralski crystal growth system (Metals Research Co). Crystal ingot is $Bi_{12}GeO_{20}$.

Single crystals of several semiconductors and optical materials obtained in our laboratory using the Czochralski, Bridgman and floating zone techniques.

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Laboratory for nanoscopy is equipped with state of art equipment for measuring the properties of materials at nano-level (Omicron variable temperature SPM and AFM, model B002645 SPM PROBE VT AFM 25 with MATRIX control system, SNOM, model TwinSNOM R).

Scanning Probe Microscope

The scanning tunneling microscopy (STM) is based on the concept of quantum tunneling. When a conducting tip is brought very near to a metallic or semiconducting surface, a bias between the two can allow electrons to tunnel through the vacuum between them. The tip to sample distance is 3-10Å. For low voltages, this tunneling current is a function of the local density of states at the Fermi level, E_f, of the sample. The variations in current as the probe passes over the surface are translated into an image. This technique is used for the characterization of the conducting and semi-conducting samples.

An atomic force microscope (AFM) was invented in 1986 by Gerd Binnig, Calvin Quate and Christoph Gerber. The AFM consists of elastic cantilever with a sharp tip (probe) at its end that is used to scan the sample surface. When the tip is brought into proximity of a sample surface, the forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law. Depending on the situation, the forces that are measured in the AFM include a mechanical contact force, Van der Waals forces, capillary forces, a chemical bonding, etc. The deflection is measured using a laser spot reflected from the top surface of the cantilever into an array of photodiodes.

The two basic modes of operation of the AFM technique are contact and non-contact mode. The distance between the tip and the sample in the contact mode AFM is 2-3Å. A repulsive interaction is dominant in this area. The reconstruction of the surface topography is done by measuring the deflection of the cantilever. In the non-contact AFM mode the tip is in the region of the attractive forces, at the distance of 10-100 nm from the sample surface. These forces are not strong enough to measure the static deflection of the cantilever directly. It is necessary to induce the oscillations of the cantilever near its resonance frequency. The interaction between the tip and the sample surface leads to frequency shifting (frequency modulation). The reconstruction of the surface topography is effected by measuring the frequency shift.

The SPM techniques have a wide range of uses in solid state physics, surface science, nanotechnology and biology.

Omicron UHV VT AFM/STM, model B002645 SPM PROBE VT AFM 25

Omicron UHV VT AFM/STM Microscope

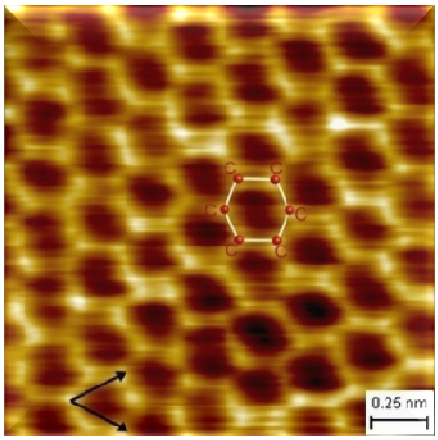
SPM head

Technical data

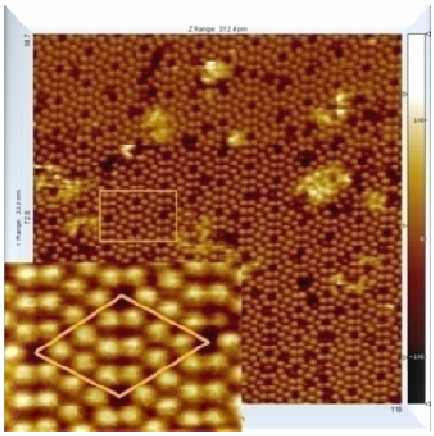
- Matrix control system
- Three modes of operation:
  - Contact mode AFM
  - Non-contact mode AFM
  - STM

  These modes provide force/distance and current/voltage spectroscopy measurements. The STM mode provides an atom manipulation facility.
- Scan (and offset) range X/Y/Z: 10µm x 10µm x 1.2µm
- Coarse movement X/Y/Z: 10mm x 10mm x 10mm; Step size: 40nm – 500nm
- Z-resolution: 0.1Å
- Measurements with atomic resolution
- Vibration isolation: Internal eddy current dumping
- Tunneling current: < 1pA – 300nA
- Gap voltage: ±5mV to ±10V; applied to tip/cantilever, sample grounded
- Ultra High Vacuum (UHV) chamber: 10^{-9}-10^{-11}mbar
- Working temperature range: 25K - 750K; with LHe or LN_2 cryostat
- Sample heating in preparation stage through direct or radiative methods up to 1000K

Some results



Highly oriented pyrolitic graphite (HOPG), atomically resolved, AFM contact mode.



Atomically resolved 7x7 reconstruction Si(111) (new surface atom rearrangement obtained by heating the sample to high temperatures under UHV conditions), STM mode.

TwinSNOM system

The TwinSNOM system consists of a room-temperature and air-condition Scanning Near-Field Optical Microscope (SNOM) and an Atomic Force Microscope (AFM). The optical microscopy is limited in resolution by the diffraction barrier. The SNOM overcomes this resolution barrier and significantly increases the resolution of the optical microscopy. For a SNOM operation a tapered and metal coated optical fiber is used. The fiber aperture diameter is much smaller than the optical wavelength and this size determines the resolution of the obtained image. In this case, there is no field propagation so the light from the fiber aperture is evanescent and the fiber tip should be brought into the optical near-field of the sample surface. The fiber tip is held in position at the focus of the optics (the reflection objective) for light detection. The sample is then moved with a scanning

motion to perform the imaging. A negative feedback loop should be used in order to control the tip to sample distance.

Omicron TwinSNOM system

The TwinSNOM is designed around a stable universal microscope stage equipped with a mechanically decoupled Zeiss Axiotech Vario microscope. The upright microscope is used for reflection mode SNOM and control of tip positioning. The shear-force AFM technique is used to solve the problem of distance regulation. In addition, it provides topographical AFM images together with every SNOM image.

The SNOM fiber tip and the shear-force detector are integrated into a single magnetically mounted and easily exchangeable sensor module. A precise positioning is provided by the use of piezoelectric stepper motors. These motors are used for the remote controlled positioning of the microscope table as well as for the precise positioning of the sensor unit at the focus of the objective. The scanner unit is integrated into the microscope table.

The SNOM control unit contains the laser and the electronics required for light detection. It includes the signal conditioning for the photomultiplier detector as well as a video input selector for the sensor approach monitoring. A highly efficient light collection is achieved in the reflection mode by a specially designed reflection objective.

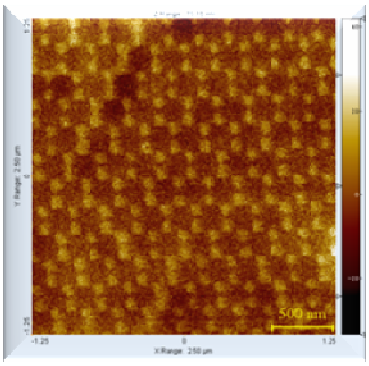
The TwinSNOM system is placed on a vibrationless table in order to protect the system from the surrounding mechanical vibrations. The table is floated using the air from a high-pressure cylinder.

The needle sensor completes the functionality of the TwinSNOM. The needle sensor allows for a high resolution non-contact mode atomic force microscopy to be used.

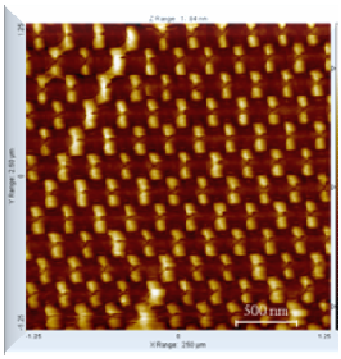
Technical data

Sample scan: lateral: 100 x 100 μm^2, vertical: 20 µm, capacitive x/y/z linearisation, 1 nm resolution.
***Sample positioning*:** 30x30 mm^2, remote controlled precision piezo drives, step size down to 70 nm.
Tip positioning: remote controlled x/y/z piezo drives, 15x15x10 mm^3, step size down to 50 nm.
***Aperture diameter*:** 50 nm nominal.
***Shear-force resolution*:** z: 1 nm, x/y: 10 nm,
Laser: current maximum: 50 mA, max. operating temperature: 50°C, max. radiation flux: ≈1 mW, emission wavelength: 635 nm
***Optical microscope*:** objectives: 50x, 10x (long distance), 10x binocular, conventional mode: upright (bright/dark field), spectral range: 350 nm to 750 nm, detection path: 1700 nm.
Footprint: 55 cm x 55 cm; 60 kg.

Some results



Fiber sensor. The sample is latex projection pattern. SNOM image.



Topography obtained by the shear-force AFM technique.

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Laboratory for the Fourier transform Infrared (FTIR) spectroscopy and ellipsometry is equipped with two FTIR systems: a Bomem DA-8, and a SPECAC spectrometers. The former system allows for the measurements in the wide frequency range (30-25000 cm^{-1}) at temperatures between 4 and 300 K. The latter set-up provides the excellent conditions for the measurements to be conducted in the low-frequency region (1-250 cm^{-1}) within the 77-300 K temperature range.

The High Resolution Variable Angle Spectroscopic Ellipsometer (SOPRA GES5E - IRSE) can measure the dielectric functions of different materials and thin films in the wide spectral range from 190 nm to 28 μm at temperatures between 10 K and 400 K using ARS Inc. low vibration closed cycle cryostat, Model CS204SE-X20(OM).

Bomem DA8 Fourier-transform spectrometer

The Bomem DA8 is a research grade Fourier-transform spectrometer for the range 4 to 50 000 cm^{-1}. The term 'research grade' refers to an instrument operating under the vacuum, having high resolution, high scanning stability and an access to several input-output ports for several different experiments. It has a vertical conventional Michelson interferometer with a patented dynamical alignment system keeping the exact alignment of the mirrors during each scan. The average angular deviations from an optimal alignment are less than 10^{-6} radians in normal laboratory environments and about 10^{-5} radians under severe vibration conditions. A special advantage of the instrument is a unique far infrared Hypersplitter that covers a broad spectral range from 40 to 700 cm^{-1}.

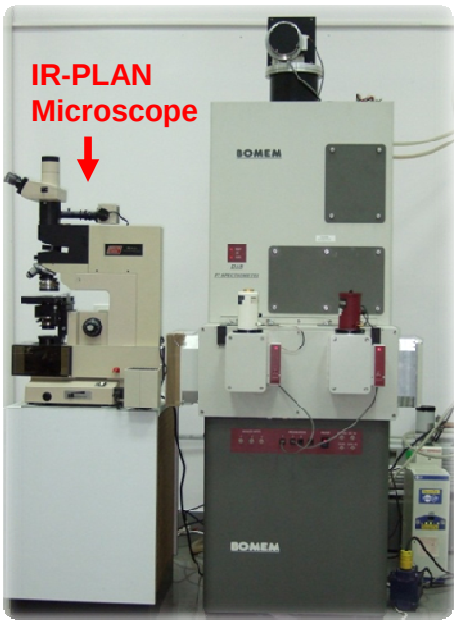


Fig. 1. The Bomem DA8 spectrometer (to the right) with the InSb and Si detectors and Spectra-Tech IR-PLAN advanced analytical microscope mounted to the left port.

The configuration of the instrument is given in Fig. 2. It consists of three sections: the upper one containing the source and the beamsplitter compartment, the middle section with the beam switching compartment, the sample compartment and the detector modules, and the lower part containing the vacuum leads, the power supplies and the data processing and control electronics. Depending of the moving mirror travel, the resolution ranges from 0.06 to 0.0026 cm^{-1}. The instrument has two focused output beams in a sample compartment and three parallel output beams as shown in Fig. 2. In the present configuration our DA8 system operates within the 10 to 25 000 cm^{-1} range with a maximal resolution of 0.02 cm^{-1}. As sources we use a Hg lamp, a Globar (SiC) and a Quartz lamp. The beamsplitters are a 25 μm mylar film (10-125 cm^{-1}), a Hypersplitter (40-700 cm^{-1}) KBr (500-5000 cm^{-1}) and a Quartz (4000 – 25 000 cm^{-1}). The following detectors are used: DTGS (far IR), MCT (77K, mid infrared), InSb (77K, near infrared) and Si photodiode (visible). The spectrometer is equipped with two cryostats: a He flow Janis STDA 100 (LN_2 and a LHe, 4-400 K) and an ARS DMX 20 closed-cycle low vibrational cryostat (4.5-300K) with vibration amplitudes in the nanometer range. The system was recently upgraded from far IR and MID IR to the NIR and VIS ranges, namely, a NIR LN_2 InSb detector (IPH5000L) for the range 1800-8500 cm^{-1}, a visible Si (IPH5600L) detector for the range 8500-50 000 cm^{-1} and a Quartz visible beamsplitter (IMB2100L) for the range 4000-25 000 cm^{-1}. For data acquisition and processing, the DA 8 uses the latest working version of the Bomem GRAMS/AI 7.0 software. In addition, a suitable IR-PLAN advance analytical microscope for measuring samples down to 7 μm in size is used as shown in Fig. 1.

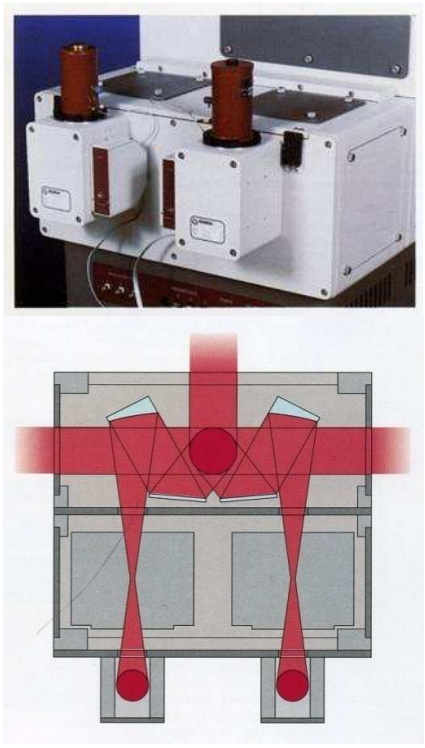


Fig. 2 The DA8 system with 5 output ports. There are two focused output beams in a sample compartment and three parallel output beams. This model is appropriate for simultaneous installation of several experiments.

Janis STDA 100 flow cryostat

ARS DMX 20 low vibration closed-cycle cryostat

Figs 3 and 4 present the polarized reflectivity spectra of the NaV_2O_5 single crystals in the far and mid infrared range.

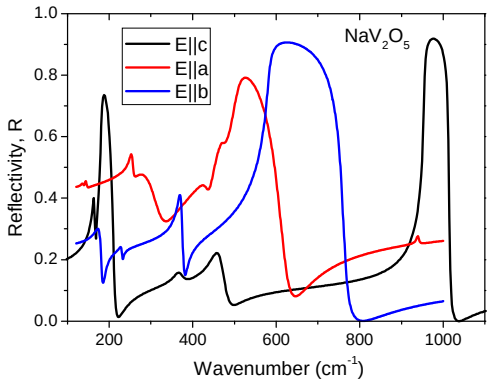


Fig. 3. Polarized FIR reflectivity spectra of NaV_2O_5

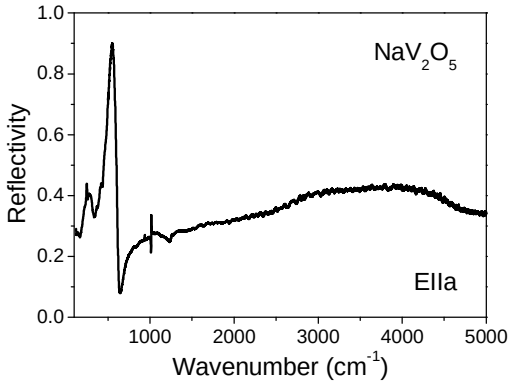


Fig. 4. E||a polarized FIR and MIR reflectivity spectrum of NaV_2O_5

IR-PLAN analytical microscope

IR-PLAN analytical microscope is a visible light microscope equipped with a high performance infrared sampling accessory designed for operation with FT-IR spectrometer. The microscope performance is directly related to the detection system because usually the analyzing samples are small so it is necessary to use MCT (mercury cadmium telluride) detector. IR-PLAN enables viewing of the exact sample area that will be analyzed and offers the best resolution available in FT-IR microscopy in order to obtain the highest possible quality spectra with the least stray light. IR-PLAN can be mounted, depending on the type of the spectrometer, in the primary compartment or in an external sample compartment or alongside of the spectrometer (Fig.1). IR-PLAN analytical microscope is equipped with a standard 1"x2" manual stage with a stage clip which provides the movement of the sample along the x, y and z-axis (focus adjustment). Usage of two circular, rotatable masking apertures above and below the sample reduces the stray light and other unwanted spectral contributions. The upper aperture is located in the infrared path between the infrared source and the sample whereas the lower aperture is located in the infrared path between the sample and the infrared detector. IR-PLAN analytical microscope can operate in **transmission** and **reflectance** mode.

Instrument specifications

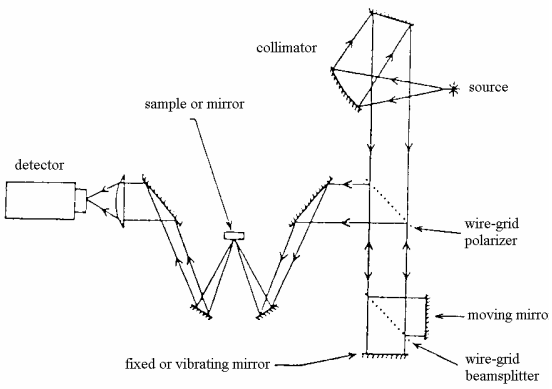
Viewing optics: Standard 15X Reflachromat IR/VIS objective of the Cassegrainian type design for 150X viewing and 10X D-plan achromat glass objective for 100X viewing and visual identification of the sample area with a larger field of view.
Viewing attachments: Binocular viewer has paired 10X eyepieces with crosshair measuring reticle. In combination with the standard 15X objective, provides over 150X visual magnification.
Detection: use of the spectrometer's detector optics and detector or a dedicated MCT detector.
Illumination: High intensity reflected and transmitted light illumination with variable light intensity and field and aperture stops.
Objective positioning: 4 position rotatable nosepiece.
Sample positioning: Standard 2"x3" travel rectangular rotatable manual stage with stage clip.
Sample masking: Two circular variable apertures for masking capable of being used simultaneously to mask the sample, anywhere in the field of view.

Field of view& sampling area: Nominal 1.3mm field of view with a 15x objective. Sampling area depend on the detector, detector optics and the collecting objective being used.
Purge: the spectrometer's purge system can be used or own purge system depending on the instrument and coupling.
Sampling mode: Transmission or reflectance.
Microscope support: Rolling work station/table.

Polarising Fourier Transform Spectrometer SPECAC 40000 for FAR IR

The instrument is designed for operation in the spectral range between 3 and 250 cm^{-1} (90 GHz to 7.5 THz). It is configured as a polarisation interferometer using a single pair of wire grid polarisers acting as a beamsplitter and operates with either a phase or an amplitude modulation in a step-scan mode.

SPECAC 40000 FAR IR spectrometer. Inset: LN_2 cryostat



Configuration of SPECAC 40000 spectrometer for reflectance measurements

The interferometer is based upon the polarising wire grid configuration developed by Martin and Puplett. The wire grids that are made from 5 μm diameter Tungsten wire spaced 12.5 (25) μm centre-to-centre. The first grid acts as a polariser to the collimated beam from the quartz-encapsulated mercury vapour arc lamp, producing two orthogonally plane polarised beams. The wires of the second grid are oriented at an angle of 45^o relative to the first one, acting effectively as a beamsplitter.

The moving mirror has a mechanical path of 50 mm which in the case of a two-sided interferogram, gives the best resolution of 0.4 cm^{-1}. The instrument is assembled with either a transmission or a reflection module for measurements on solid state specimens.

An advantage of this spectrometer is a phase modulation which has a better signal-to-

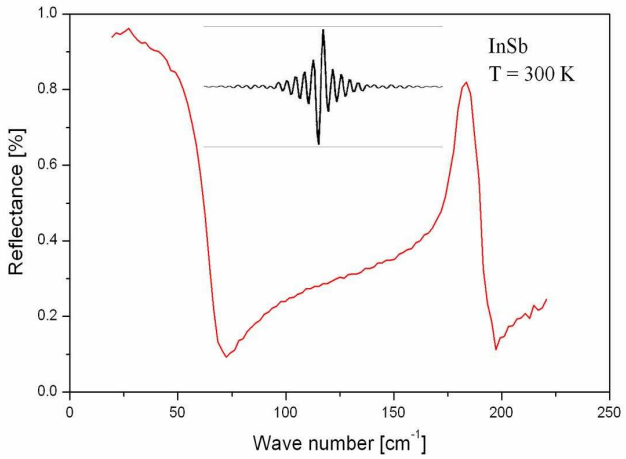
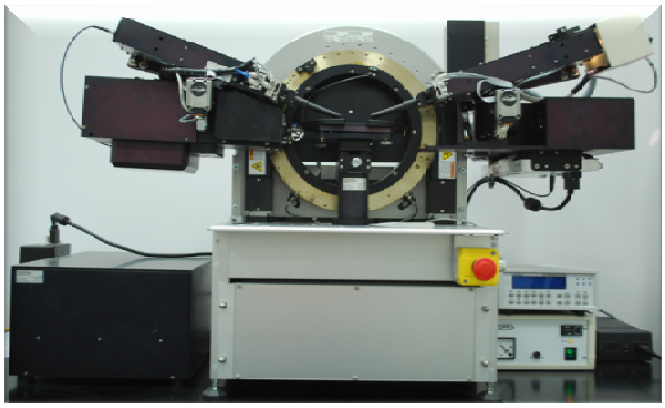


Fig.5. FIR reflectivity spectrum of InSb measured using Specac system at 300 |K

noise ratio comparing to amplitude modulation. In the case of the phase modulation the fixed mirror vibrates with the frequencies between 10 and 20 Hz. As a consequence we have an asymmetric interferogram with the zero-leveled background as it is shown in the inset of Fig. 5. For both modulations a lock-in amplifier must be used. The instrument works under the vacuum and is equipped with a LN_2 cryostat. In Fig. 5 the reflectance spectrum of InSb is presented.

GES5E-IRSE Variable Angle Spectroscopic Ellipsometer

The GES5E-IRSE Spectroscopic Ellipsometer is a combined system consisting of: DUV-Visible-NIR Spectroscopic ellipsometer (SE) and Fourier Transform Infra-Red Spectroscopic Ellipsometer (FTIR-SE). The polarizer and analyzer arms of both ellipsometers are mounted on a high resolution goniometric bench, made of double hollow crown. Both these crowns are driven by computer controlled stepper motors. The incidence angle can vary from 7 to 90° in DUV-Visible-NIR range, and from 20 to 90° in MIR range, with a theoretical resolution of 0.0005°.



GES5E-IRSE Spectroscopic Ellipsometer

The DUV-Visible-NIR Spectroscopic ellipsometer (SE) is working in a rotating polarizer configuration. It operates on the principle of mechanical modulation of the incidence light polarization by rotation of the polarizer at a constant angular frequency of 9 Hz. The analyzer remains in a fixed position, preparing the signal for the detector, insensitive to polarization. The precision of the ellipsometer for both weak and strong absorbing materials characterization is significantly enhanced with an automatically adjustable compensator. The light source is one 75 W Xe arc lamp, directly adapted to the polarizer arm. It emits a continuous spectrum of light, ranging from ultraviolet, trough visible to infrared (185-2000 nm). The light spot on the sample in parallel beam configuration is 1-10 mm^2, depending on the aperture. There is also an additional miscrospot option for focusing the beam with a spot size of 365 x 270 μm, for an incidence angle of 75°. The light is introduced from the analyzer arm to the spectrometer using optical fiber making the light beam more stable. By combining the spectrometer with the photomultiplier tube (PMT) detector in UV-VIS range (190-900 nm), and InGaAs detector in NIR range (750-2000 nm), a high resolution spectrum is obtained by scanning the ellipsometric image at many discrete wavelengths. The spectrometer contains two dispersive elements (grating and prism) in collaboration with each other, separated by an intermediate fixed slit. The grating is blazed at optimum wavelength in order to obtain a maximum efficiency and the prism refracts the incoming wavelengths to act as a filter for higher order of diffraction produced by the grating.

The Fourier Transform Infra-Red Spectroscopic Ellipsometer (FTIR-SE) is a combination of a rotating analyzer configuration and a Fourier transform spectrometer. The basis of the FTIR spectrometer is a Michelson interferometer, which modulates each wavelength by a different frequency. The light leaving the Michelson interferometer enters the ellipsometer and successively passes the polarizer, the sample, the analyzer and finally hits the detector.

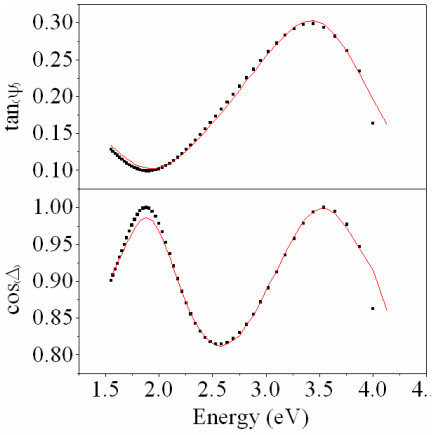
The light source for this IR ellipsometer is a silicon carbide (SiC) globar. Our system uses two different detectors: MCT in a range 580 cm^{-1} to 7000 cm^{-1}, and DTGS in a range 385 cm^{-1} to 6500 cm^{-1}. There is also an optional compensator in order to improve the accuracy of the measurements.

The ARS DX204 closed cycle low vibration cryostat with additional DMX-20 interface allows for the low temperature measurements up to 4.5 K. This cryostat is mounted on a specially designed SOPRA stage and SOPRA chamber with three possible angles of incidence (60°, 75°, 90°).

The spectroscopic ellipsometry measuresthe change in the polarization of light upon oblique light reflection on the surface of the sample to be studied. The incident light is linearly polarized, in general, it becomes elliptically polarized upon reflection. The polarization state of incidence light can be decomposed into *s*- and *p*- components, one perpendicular, and the other parallel to the plane of incidence. These two components reflect in a different manner, depending on the reflection properties of the surface. The ellipsometer measures ψ and Δ, which represent the ratio of reflection coefficients r_p and r_s, written as: $\rho = r_p/r_s = \tan(\Psi)e^{i\Delta}$. Thus, $\tan(\psi)$ is the amplitude ratio upon reflection, and Δ is the phase difference.

The spectroscopic ellipsometry can provide information about very thin layers, even down to a single atomic layer, or less. The measurements of the complex refractive index or dielectric function tensor give access to fundamental physical parameters that are related to a variety of sample properties, that include morphology, crystal quality, chemical composition, or electrical conductivity. It is commonly used to characterize single layer thin films or complex multilayer stacks ranging from a few parts of nanometers to several micrometers with an excellent accuracy.

ARS DX204 closed cycle cryostat with SOPRA vacuum shroud.



Measured ellipsometric parameters (circles) of thin films on a glass substrate and the best fit (full line)

Best-fit model sketched

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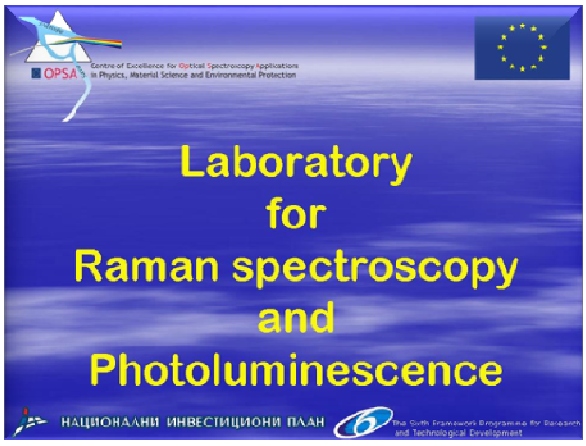
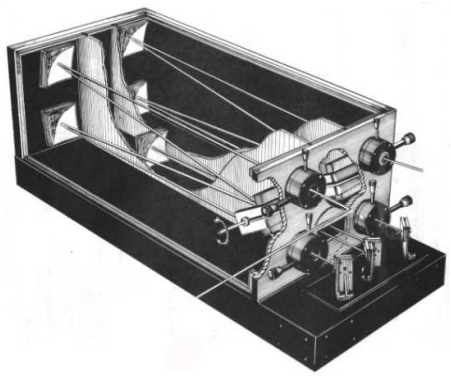


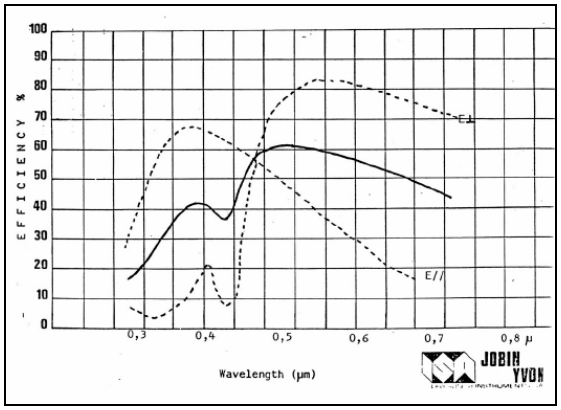
Laboratory for Raman scattering spectroscopy and photoluminescence is equipped with double grating U1000 Jobin Yvon monochromator, Ar, Kr, He-Ne and He-Cd ion lasers, and Peltier effect cooled photomultiplier (model RCA 31034A) as a detector (single photon counting detection system). For low-temperature measurements (10 K-400 K) there is the Leybold closed cycle helium cryostat. This experimental set-up has an excellent stray-light rejection and allows for the measurements close to the laser line to be made.

Jobin Yvon U1000 Raman spectrometer

The main part of the Raman scattering spectroscopy laboratory is the ***Jobin Yvon U1000 double monochromator***, which contains two holographic ***1800 grooves/mm gratings***, whose synchronized rotation leads to the light dispersion. The U1000 double Raman spectrometer has a long 2 x 1 m focal length with a high precision drive mechanism. The double additive mode of the monochromator is ideally suited to *very high spectral resolution* (*about 0.15 cm^{-1}* at 579.1 nm - Hg line) and very high stray light rejection (10^{-14} at 20 cm^{-1} from the Rayleigh line) applications, allowing the collection of *low-frequency Raman spectra down to* 2-5 cm^{-1}. The dispersion of the monochromator is 9.2 cm^{-1}/mm (0.243 nm/mm) for 514.5 nm, whereas its quantum efficiency is greater than 40% in the range (440–720) nm.



Jobin Yvon U1000 double monochromator



Quantum efficiency of U1000 gratings.