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.

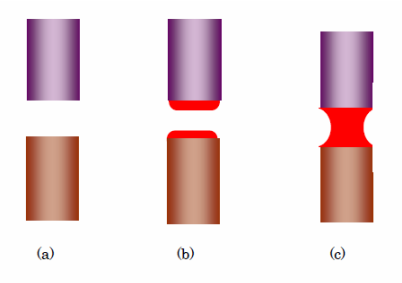
*Four mirror optical furnace model FZ-T-1000-H-HR-I-VPO-PC (Crystal System Co.) with vertical molysili furnace model VF1800 (right).*

**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**

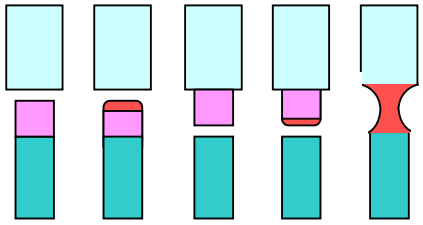



(a) At the initial stage of the heating, both the feed and seed rod are set apart by a distance of a few mm;

(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**



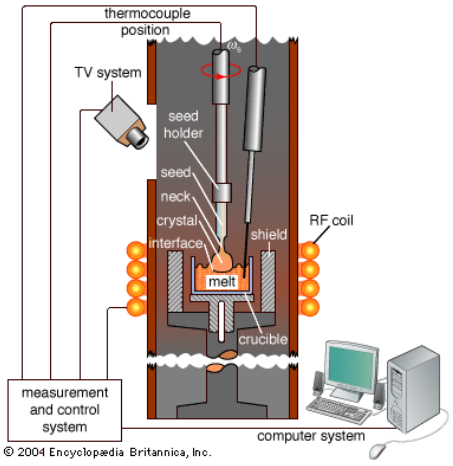
1) **First stage**: Set the solvent zone chip on the top of the seed rod. A small amount of starch paste can be used to fix it. The starch will be burnt off as the temperature increases, and a small amount of smoke will appear.
**2) Second stage:** Both the top of the solvent zone chip and the end of 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,

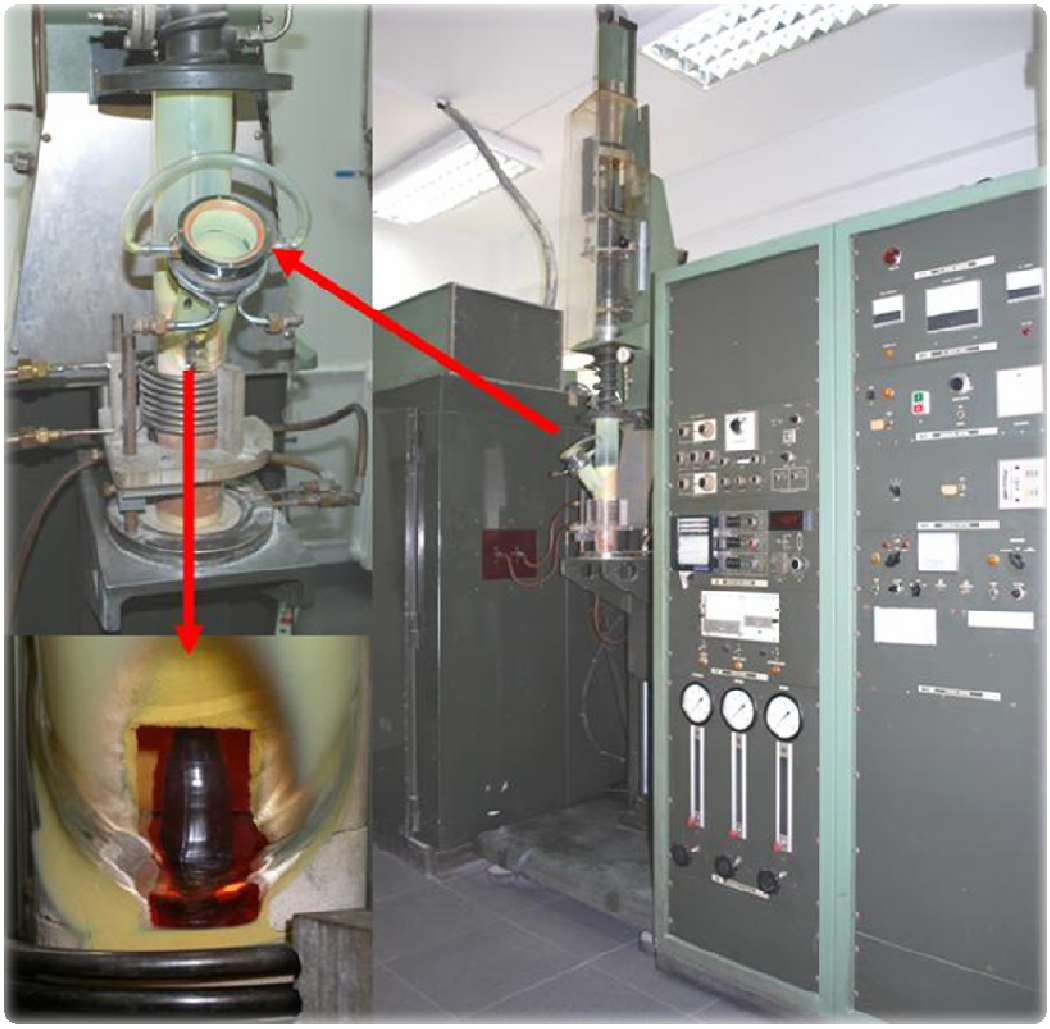


gold), salts and some hand made, (or "lab") gemstones.

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*

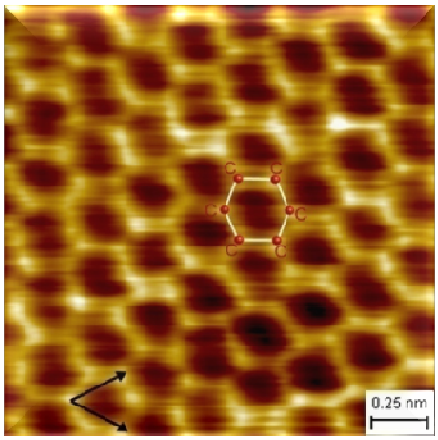
*SPM head*

### Technical data

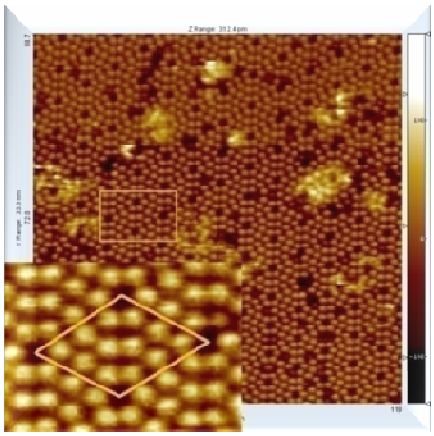
- Matrix control system
- Three modes of operation:
  - Contact mode AFM
  - Non-contact mode AFM
  - STM

  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.

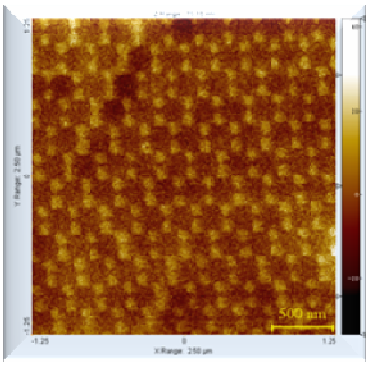
The TwinSNOM system is placed on a vibrationless table in order to protect the system from the surrounding mechanical vibrations. The table is floated using the air from a high-pressure cylinder.

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.

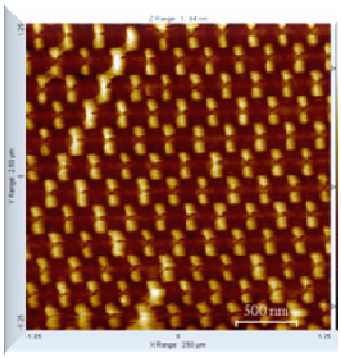
## Technical data

***Sample scan***: lateral: 100 x 100 $\mu m^2$, 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 $cm^{-1}$.

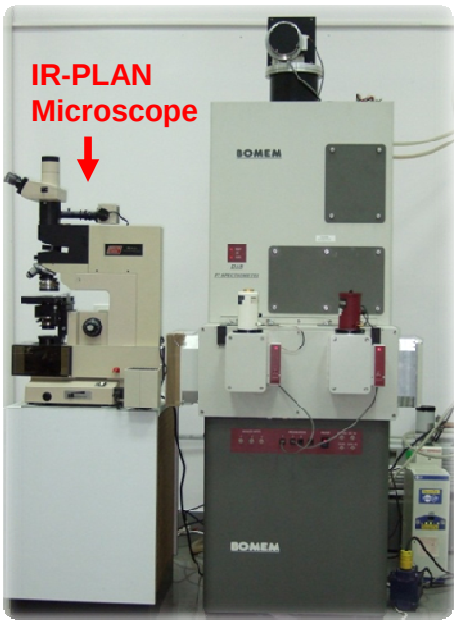



*Fig. 1. The Bomem DA8 spectrometer (to the right) with the InSb and Si detectors and Spectra-Tech IR-PLAN advanced analytical microscope mounted to the left port.*

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.

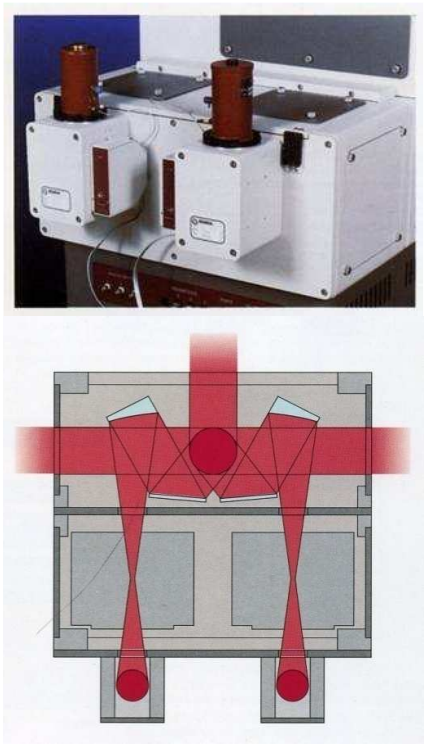


*Fig. 2 The DA8 system with 5 output ports. There are two focused output beams in a sample compartment and three parallel output beams. This model is appropriate for simultaneous installation of several experiments.*

*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.

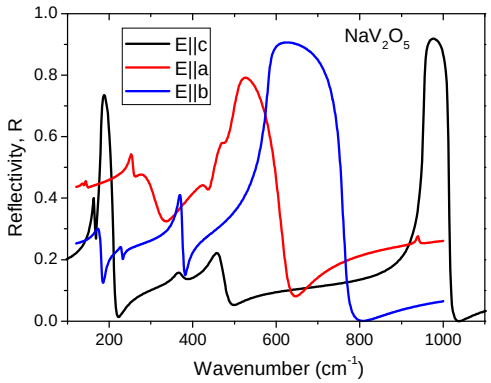



*Fig. 3. Polarized FIR reflectivity spectra of $NaV_2O_5$*

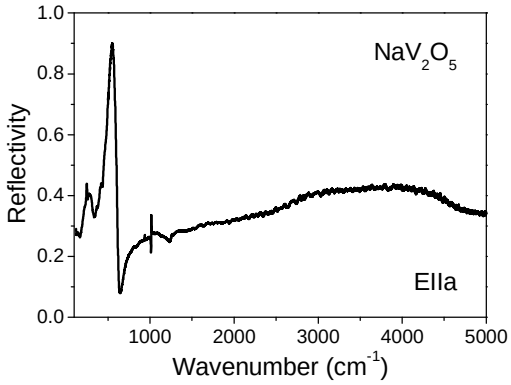



*Fig. 4. E||a polarized FIR and MIR reflectivity spectrum of $NaV_2O_5$*

## 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

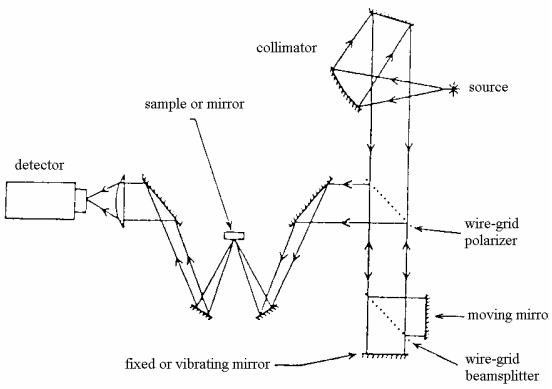
**Viewing optics**: Standard 15X Reflachromat IR/VIS objective of the Cassegrainian type design for 150X viewing and 10X D-plan achromat glass objective for 100X viewing and visual identification of the sample area with a larger field of view.
**Viewing attachments:** Binocular viewer has paired 10X 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 $\mu$m diameter Tungsten wire spaced 12.5 (25) $\mu$m 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-

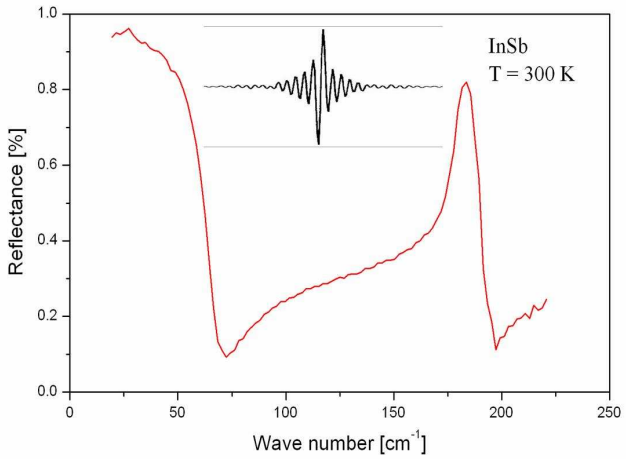
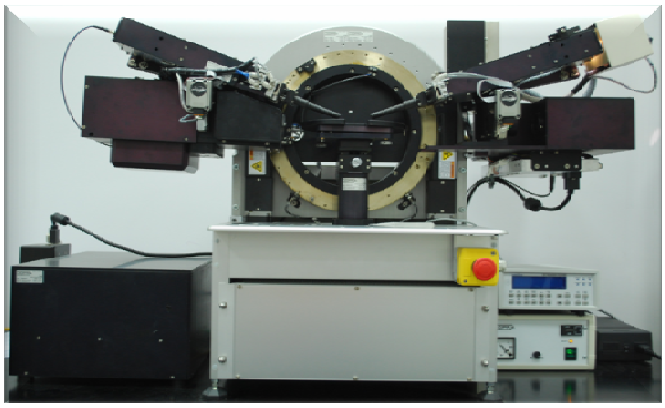



*Fig.5. FIR reflectivity spectrum of InSb measured using Specac system at 300 |K*

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.

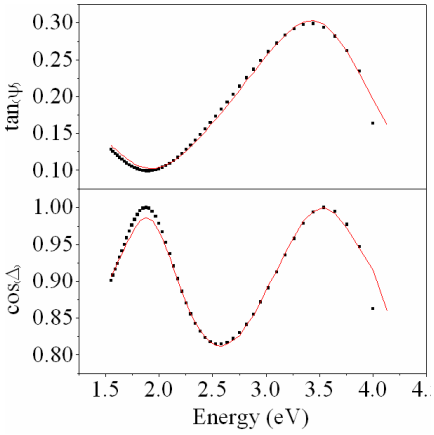
The light source for this IR ellipsometer is a silicon carbide (SiC) globar. Our system uses two different detectors: MCT in a range 580 $cm^{-1}$ to 7000 $cm^{-1}$, and DTGS in a range 385 $cm^{-1}$ to 6500 $cm^{-1}$. There is also an optional compensator in order to improve the accuracy of the measurements.

**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 $\psi$ and $\Delta$, 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 $\Delta$ 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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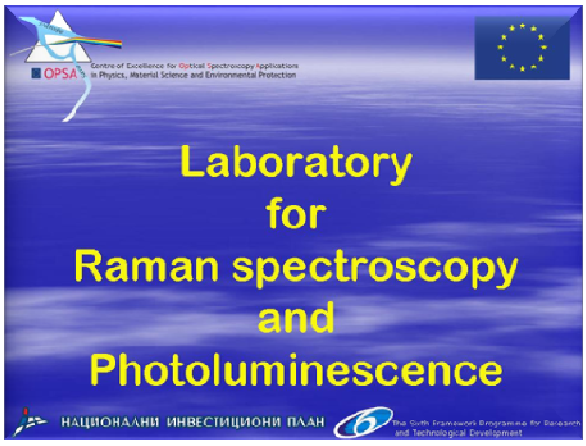
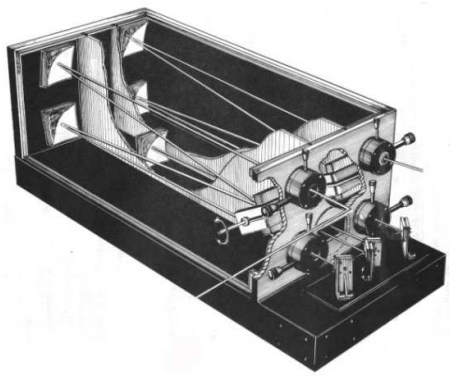
* *



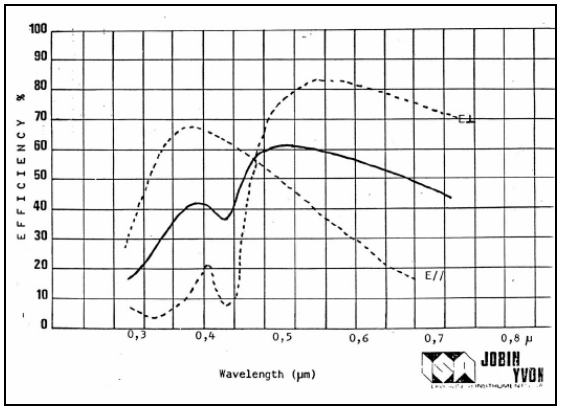

**Laboratory for Raman scattering spectroscopy and photoluminescence** is equipped with double grating U1000 Jobin Yvon monochromator, Ar, Kr, He-Ne and He-Cd ion lasers, and Peltier effect cooled photomultiplier (model RCA 31034A) as a detector (single photon counting detection system). For low-temperature 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.



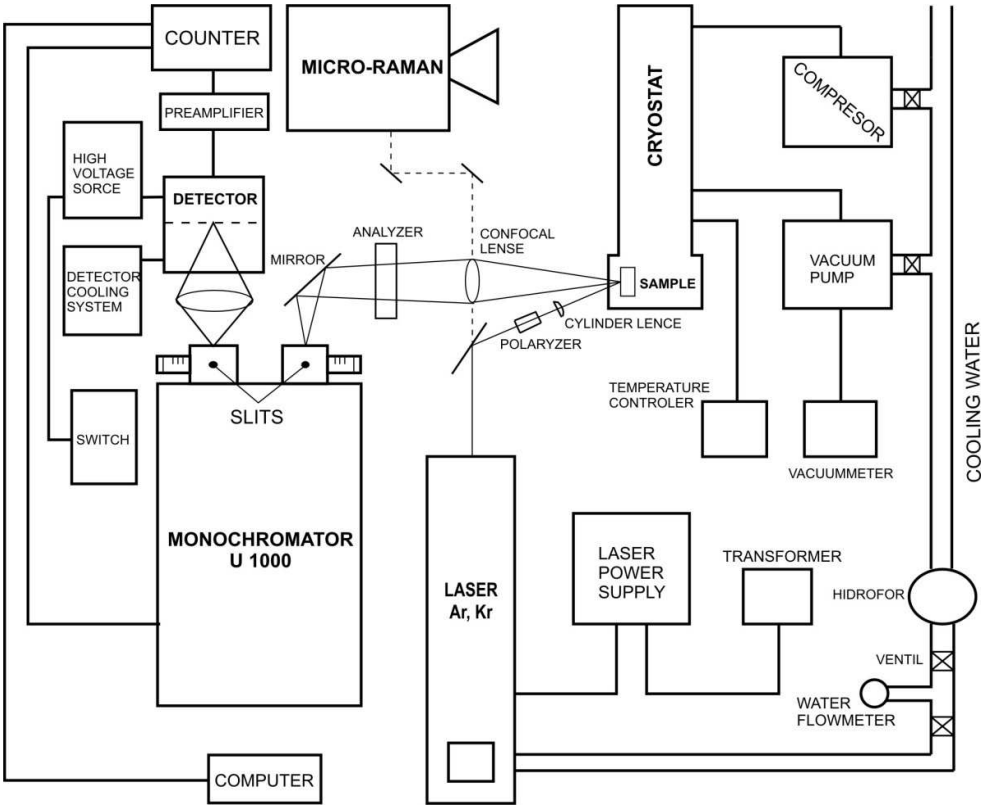
*Jobin Yvon U1000 double monochromator*




*Quantum efficiency of U1000 gratings.*

After leaving the monochromator, the light enters the detector system consisting of RCA-C31034A ***photomultiplier*** (detection system which counts photon by photon) with housing cooled by Peltier element, amplifiers and counters. ***Ar, Kr, He-Ne*** and ***He-Cd ion lasers*** are used as light sources.

A detailed scheme of the Raman scattering spectroscopy and photoluminescence experiment is shown in figure below. The laser spot is focused by the cylindrical or dichromate lens at the sample surface with a very small angle of incidence. Such configuration corresponds to the so-called quasi-backscattering geometry. The laboratory is also equipped with the helium closed cycle ***Leybold*** cooling system, which allows for the Raman scattering measurements at low temperatures down to **10 K**.




*Detailed scheme of the Raman scattering and photoluminescence experiment.*

The spectra acquisition is controlled by ***LabSpec software***, which provides for a choice of measurement spectral range, step and integration time in each step, by taking into account a laser wavelength. An illustration of the Raman spectrum *(excited by 442 nm line of He-Cd laser),* measured in 4 regions with different resolution is presented in figure 6.

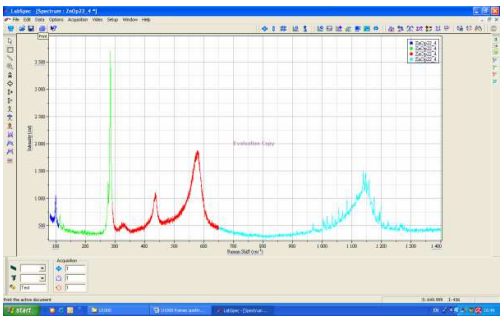
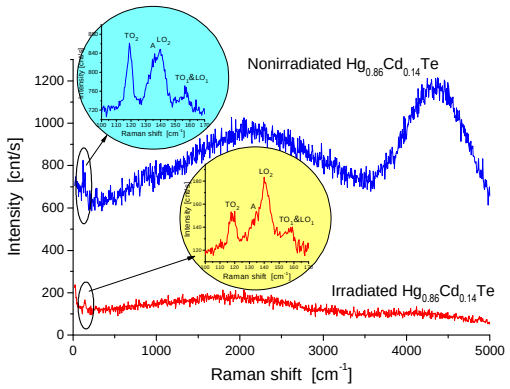


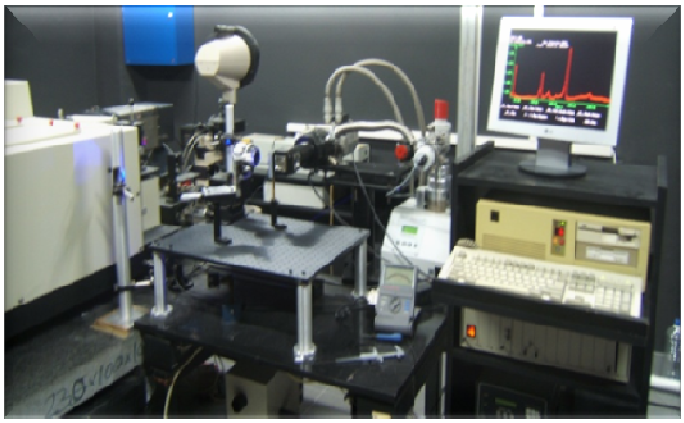
*Figure 6. Raman spectrum of mechanically activated ZnO powder.*

The U1000 double monochromator allows for simultaneous measurements of the Raman and the photo-luminescence spectra. As a rule, the photoluminescence spectra are measured with a lower resolution and a wider spectral range compared to the Raman spectra. The spectral range of the instrument depends on laser

wavelength (max range is 350-850 nm).




*Raman (insets) and photoluminescence spectra of $Hg_{0.86}Cd_{0.14}Te$ single crystal*



*Raman scattering experiment*

*

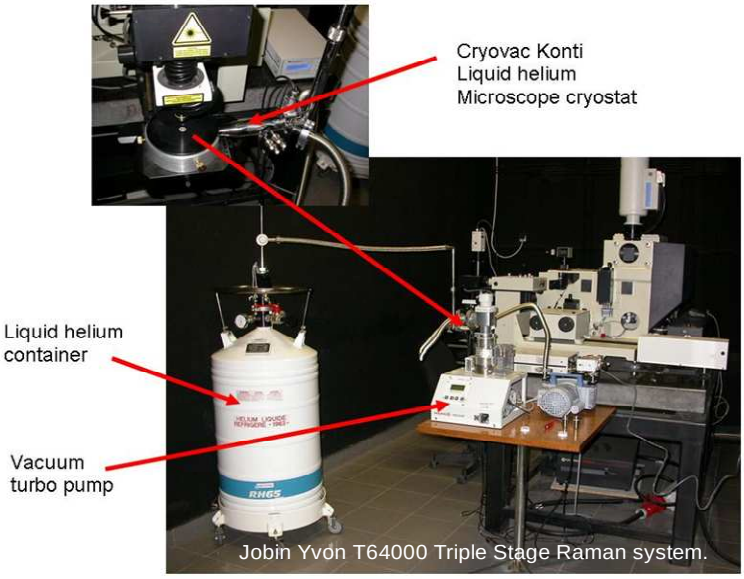
* *


**Laboratory for micro-Raman scattering spectroscopy** is equipped with triple Jobin Yvon T 64000 spectrometer (gratings with 1800 grooves/mm), a Coherent Ar- Kr mixed gas ion laser, and a CCD detection system. The set-up contains a confocal microscope and an x-y-z microscope stage. For variable temperature measurements there is a Linkam THMS 600 heating and cooling microscope stage, which allows for the micro-Raman measurements to be performed within the 77 to 900 K temperature range. Another micro-Raman set-up is a TriVista 557 spectrometer. A Konti liquid helium microscope cryostat is also part of this laboratory.

## Jobin Yvon T64000 Raman spectrometer

The Jobin Yvon T64000 Raman system is equipped with a ***triple Jobin Yvon T64000 spectrometer, Coherent mixed $Ar^+/Kr^+$ ion gas laser*** and ***CCD detector system***. The experimental set-up also includes a ***confocal microscope and*** a ***XYZ stage***. The ***Cryovac Konti cooling cryostat***

for low temperature measurements makes it possible to obtain the micro-Raman spectra within the temperature range **from 4 to 300 K**. Through appropriate neutral filters of the T64000 system, the laser spot is driven by the mirror combination to the microscope system, which focuses a spot on the sample surface. The ***confocal microscope*** permits experiments on small size samples, with improved spatial, lateral and depth resolutions. The light scattered from the sample surface is redirected to the microscope and than orientated by mirrors to the triple monochromator. The spectral dispersed light from the monochromator enters the ***CCD (Charge Coupled Device) detector***.




*Jobin Yvon T64000 Raman scattering system with Cryovac Konti microscope cryostat.*

The T64000 monochromator system consists of a ***double pre-monochromator*** (stage 1 and 2, see figure 7) and a ***spectrograph*** stages (stage 3 in the same figure). The pre-monochromator is a twin monochromator working in a subtractive mode. It acts as a tunable filter in the spectral range defined by the scanning mechanism and the gratings. The spectrograph stage is used as disperser. The T64000 system can be utilized in a ***triple additive*** or in a ***triple subtractive modes***. Its unique optical design allows for easy switching between additive and subtractive modes. An additive mode gives *the highest spectral resolution* and high linear dispersion, whereas a subtractive mode gives a *high stray light rejection* and allows for collecting *low-frequency Raman spectra down to* 5 $cm^{-1}$. The optical diagrams for these two modes are shown in figure 7.

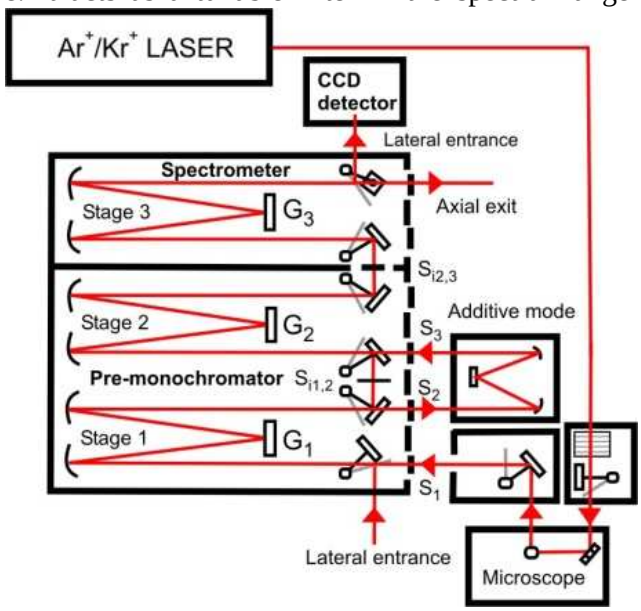



*Fig.7. Optical diagram of JY T64000 monochromator in a triple additive and subtractive mode.*

In the triple subtractive mode, which is used more often than the additive one, a polychromatic radiation enters the first monochromator through the entrance slit $S_1$ and is dispersed by the grating $G_1$. The exit slit of the first monochromator (entrance slit

of the 2nd monochromator) $S_{i1/2}$ selects a bandpass between $\lambda_1$ and $\lambda_2$. The grating $G_2$ in the 2nd monochromator recombines all the dispersed radiations on the exit slit $S_{i2/3}$ giving again a polychromatic radiation, but limited to only the spectral range between $\lambda_1$ and $\lambda_2$. In that manner, the elastic radiation remains outside this range, which is the main purpose of the pre-monochromator. The polychromatic radiation selected by the pre-monochromator is dispersed by the grating $G_3$ of the spectrograph and is directed by mirrors to the lateral exit of the spectrograph. The T64000 is equipped with three holographic ***1800 grooves/mm*** gratings defining a mechanical range of ***0-950 nm***.

***The Symphony 1024 x 256 Cryogenic Open Electrode CCD detector***, cooled by nitrogen down to 140 K, is mounted in the plane of the exit image of the T64000 monochromator. With an average quantum efficiency of 40% from 200 nm to 900 nm and its relatively flat response (Fig.8), this detector is the optimal choice for general purpose spectroscopic measurements.

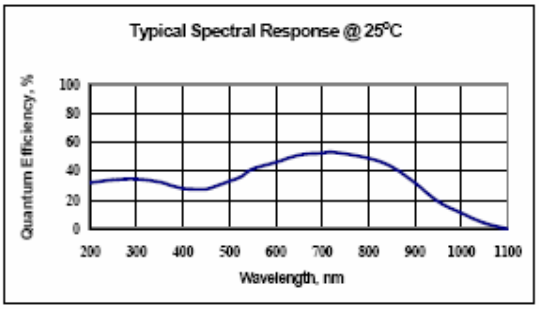



*Fig.8. Quantum efficiency of CCD detector.*

The processes of measurement and acquisition are controlled by a ***LabSpec software***. With regard to the efficiency of its detection, the T64000 system makes it possible to gather weak signals. The exposure time and the accumulation number are chosen in order to obtain the best signal to noise ratio.

***System technical specification:***

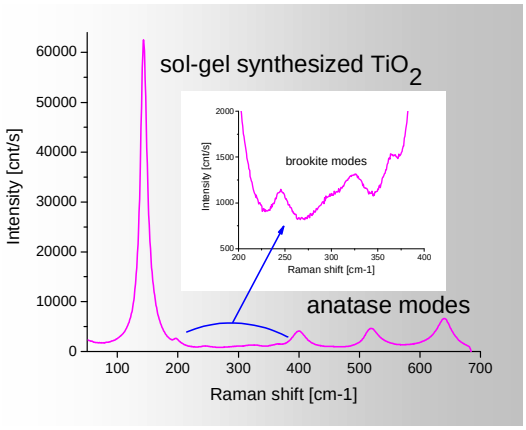
**Focal length:** 3 x 640 mm (triple additive)

**Low frequency:** typically 2 – 5 $cm^{-1}$ (double filter stage)
**Stray light rejection:** $10^{-14}$at 20 $cm^{-1}$ (514 nm laser)
**Resolution:** better than 2 pixels of the CCD, i. e. 2 $cm^{-1}$ (for 514,5 nm laser)
**Dispersion**: 17,68 $cm^{-1}$/nm (at 600 nm)

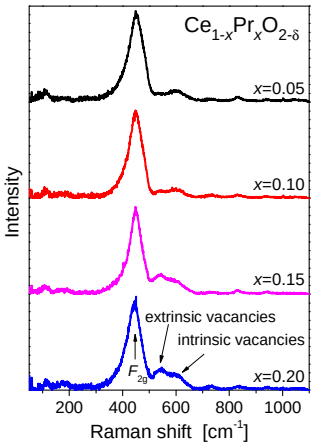
**Step size:** 0.00066 nm (with 1800 grooves/mm gratings)
**Reproducibility:** better than 1 pixel

**Gratings:** 3 x 1800 grooves/mm
**Precision:** ±1 $cm^{-1}$ in the range (450 - 850) nm
**Spectral range:** 0 - 950 nm

Jobin Yvon T 64000 system allows for the measurements of high resolution Raman spectra to be made in a relatively short time, which makes it possible that a small content of highly disordered phase in materials be registered, as it is illustrated in the case of anatase $TiO_2$ or Pr doped $CeO_2$., see below.




*Raman spectra of anatase $TiO_2$.*
*Mode of brookite phase can be also detected*




*Oxygen vacancy modes in $Ce_{1-x}Pr_xO_2$*

## TriVista TR557 Raman spectrometer

The TriVista 557 (S&I GmbH) is a triple spectrograph which offers ***the highest spectral resolution*** and ***extreme stray light rejection*** required for the ***Raman and photoluminescence*** measurements in ***UV, VIS, and NIR spectral ranges***. Its unique optical design (patent pending) allows an easy switching between ***additive and subtractive modes*** and it can be easily reconfigured to work as a ***double or a single spectrometer***.

In the heart of the TriVista are industry leading Acton Research Corporation spectrometers. They are known for superb resolution, stray light rejection, excellent imaging and ruggedness. The TriVista can operate ***from 185 nm to 2.2μm***. The spectral ***resolution can reach 4 picometers*** in the VIS spectral range (500 nm). The extreme stray light rejection allows Raman spectra to be measured as ***close as 5 wave numbers*** from the Rayleigh line.

*TriVista 557 system (S&I GmbH)*

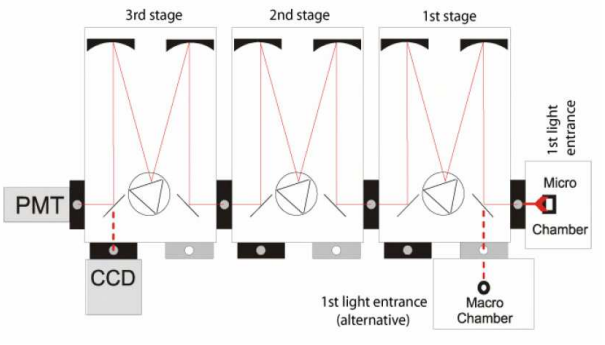



*Fig.9. Triple Raman Spectrometer in additive or subtractive mode with Macro- or Micro-Chamber and PTM and CCD detectors.*

The TriVista is the most flexible system for scientific use on the market. ***Nine gratings (three in each stage)*** with different numbers of grooves per mm (from 300 to 2400 grooves/mm) ensure the collecting of the Raman spectra in different ranges and with different resolutions just by applying software commands. The TriVista 557 model has 500 nm focal length in the first and the second stages and 750 mm in the third stage. It can be used in ***single, double and triple configurations***. A single configuration means all three stages can be used simultaneously and independently for three different experiments to be run at once. Most often the TriVista is utilized as a double or triple system. In these cases, the light beam sequentially passes through 2 or 3 stages and the gratings of the involved stages coherently move together with very high precision. Two most common reasons for using a double or a triple system instead of a single spectrometer are a high spectral resolution and a high stray light rejection. These two effects can be achieved in different modes of the TriVista operation:

1. The Additive mode gives a high spectral resolution and high linear dispersion.
2. The Subtractive mode gives a high stray light rejection.

The TriVista software offers an easy way to switch between additive and subtractive modes just by mouse-clicking. The physical mechanism behind this switch is changing the direction of the grating rotation. In the additive mode both gratings in the first and the second stages synchronously rotate clockwise adding dispersion to each other. In the subtractive mode the grating in the first stage rotates clockwise but the grating in the second stage synchronously rotates counter- clockwise precisely cancelling dispersive action of the grating in the first stage.

As it can be seen from the figure above, in triple configuration the Double monochromator stage is used together with the last stage as a Triple system for Raman spectroscopy. However, it can be also used as an excitation stage for ***Fluorescence and Photoluminescence*** and the emission can be detected by the last stage of the system.

The ***S&I software*** was written to obtain an optimized access to all three stages of the TriVista (Fig. 9). It is programmed in "Visual Basic" and runs in co-operation with the Princeton Instruments' ***WinSpec software*** package, which is designed to operate a multitude of CCD detector and allow access to exclusive detector functions. The S&I software controls spectrometer functions while the WinSpec is used as a DLL and provides data acquisition and setup functions for multi-channel detectors.

The TriVista spectrometer is equipped with the *Princeton Instruments Spec-10: 256* detector (Fig.10), which is a fully integrated spectroscopic ***CCD system***. A choice of industry standard, spectroscopic-format E2V sensors are offered. The Spec-10: 256E incorporates an open electrode sensor which offers a broadband response over a wide spectral region - from 200 to 1050 nm, as can be seen from the quantum efficiency curve in the Fig. 11. The liquid nitrogen cooling of the CCD effectively eliminates dark noise, even for long exposures.

*Fig.10. Spec-10: 256E CCD Detector*

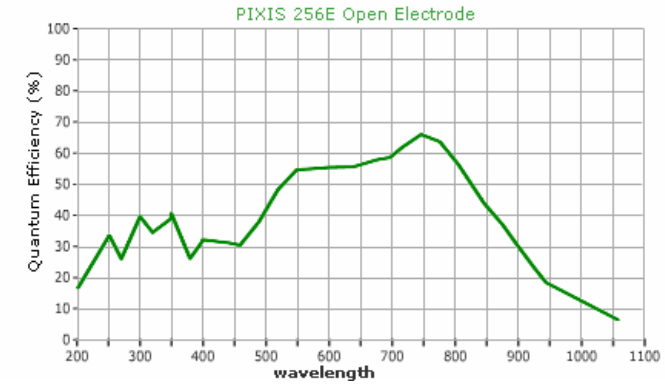



*Fig.11. Quantum efficinecy of Spec-10: 256E*

To simultaneously obtain ***Stokes and Anti-Stokes Raman spectra*** (see Fig. 12), the TriVista system is equipped by a laser mask (Fig.13) which can be installed on 1$^{st}$ , 2$^{nd}$, or on both intermediate slits. The laser mask is a very thin metal bar positioned precisely in the middle of the slit which mechanically blocks the laser light. For more versatility, the laser stop mask has 4 options - three bars of different width (150, 300, -600 μm) and the open space to allow the Raman signal to pass unblocked through the intermediate slit. The laser stop mask is set on a sliding strip for changing between 4 options and a precise positioning.

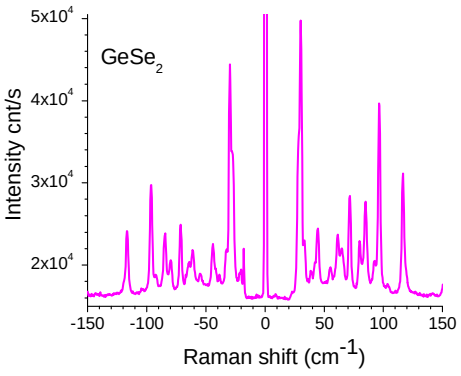
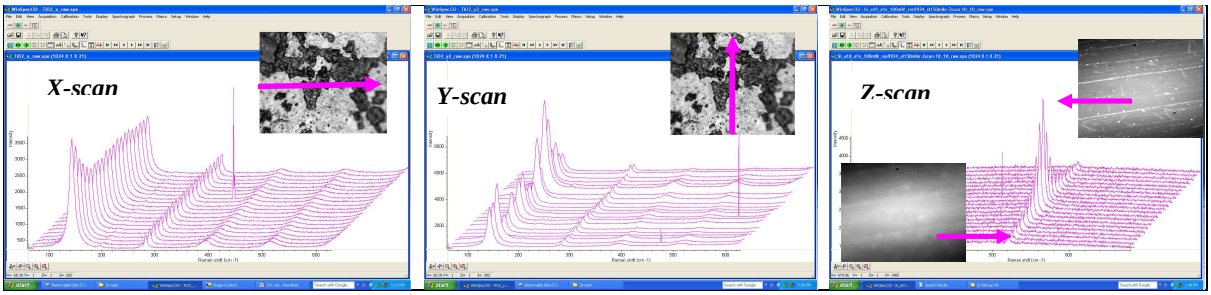



*Fig. 12 Stokes and Anti-Stokes Raman spectra of $GeSe_2$*

*Fig.13. Sliding Strip with Laser Stop Mask*

The Micro-Raman assembly is based on the upright microscope *BX51* from *Olympus* and the ***Confocal Micro-Raman Interface*** (CMRI) as an extension for the *BX51* to allow for the Micro-Raman Spectroscopy. CMRI is designed to allow direct coupling and fibre coupling for transmission of a laser beam and a Raman signal. Polarisation dependent measurements are possible for both direct coupled and fibre coupled laser beams. The ***confocal Raman microscope*** has a spatial resolution on a micron scale.

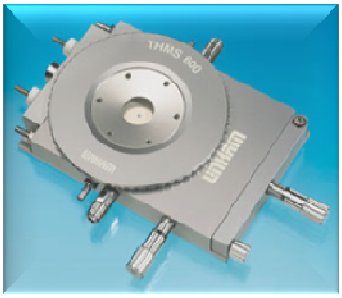
The TriVista system is equipped with a CCD detector, a confocal microscope, whereas the software-driven XYZ stage makes possible an automated 3D mapping with an auto focus option. For variable temperature measurements under microscope we have supplied Linkam THMSG600 heating/freezing stage which works in temperature range -196° to 600°C, up to 130°C/min heating and temperature stability <0.1°C.




*X-scan and Y-scan: Raman spectra of the TiO2 at various positions on the sample along x-and y-axes*
*Z-scan: Raman spectra of the Si thin film at various positions on the sample along z-axis;*

**LINKAM THMS600 - System description**

The THMS600 is one of the most popular heating and freezing stages used in many applications where high heating/freezing rates and 0.1°C accuracy and stability are needed. The LINKAM THMS600 heating/cooling stage is equipped with a CI94 temperature controller and a LNP94 Liquid Nitrogen Pump.



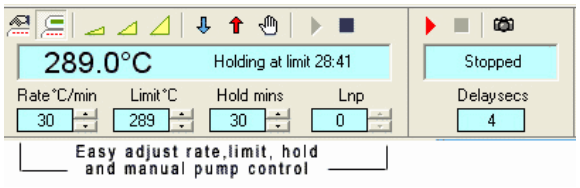

*LINKAM THMS 600 heating/ cooling stage*

*CI94 temperature controller*

*LNP94 Liquid Nitrogen Pump*

**Technical data:**

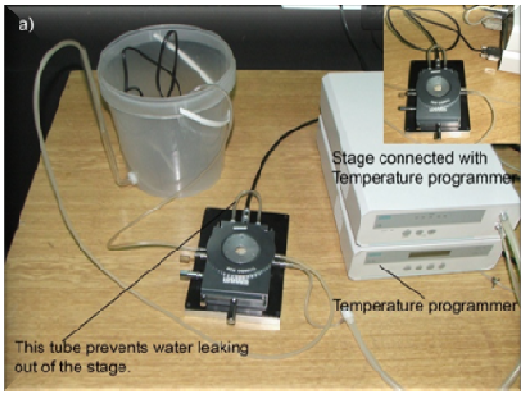
- Temperature range -196° to 600°C.
- Up to 130°C/min heating.
- Temperature stability <0.1°C.
- 16 mm X,Y sample manipulation.
- Sample area 22 mm diameter.
- 100 Ω platinum resistor sensor.
- Light aperture - 2.4mm Ø.

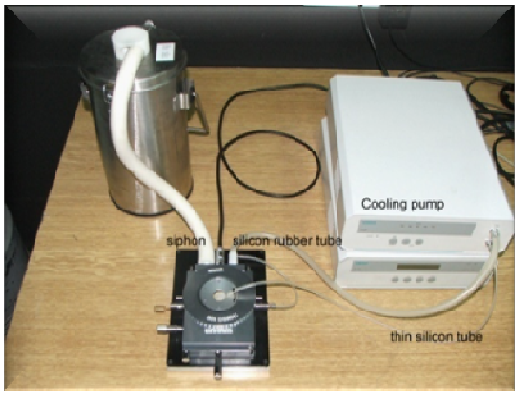



*The Linksys 32 software displays the live temperature, active ramp information and allows for the user to have full control of the rate of heating, limit and hold time*

- Silver heating block for high thermal conductivity.
- Direct injection of the coolant into the silver block.
- Single ultra thin lid window - 0.17mm.
- Objective lens working distance - 0.1mm to 4.5mm.
- Water cooled stage body for high temperature work (>300°C).
- Sample side loading without removing the stage lid.
- Can be used with all microscope techniques
- Controlled heating rates of 0.01°C to 130°C/min.
- Controlled cooling rates of 0.01°C to 100°C/min
- Displays Temperature to 0.01°C
- Hold time 0 - 9999 mins.
- RS232 interface to allow programming by Linksys software.
- A cooling system consists of the 2 Litre dewar and a control unit housing the pump which can be controlled by selecting one of 5 manual pump speeds.
- Recycled dry nitrogen gas is used to purge the sample chamber and upper lid window surface of condensation.
- The precise control of liquid nitrogen flow allows for specific stages controlled cooling rates as fast as 130$^{O}$C/min or as slow as 0.1$^{O}$C/min.

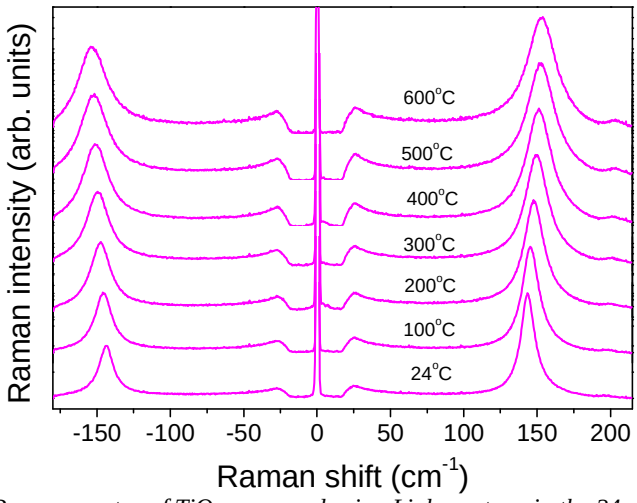
## Heating / Cooling Procedure




***Heating Procedure***
*The dewar with a pump inside should be approximately 2/3 filled with water. The heating is provided using Linksys32 software.*

***Cooling Procedure***
*The dewar should be 2/3 filled with liquid nitrogen.The lid must be placed so that the thin capillary tube is pointing upward. The thin silicon tube carries exhaust nitrogen gas and is used to prevent the blurring of the top of the window on the stage lid.*




*Stokes and anti-Stokes Raman spectra of $TiO_2$ measured using Linkam stage in the 24 – 600 $^{o}$C temperature range.*

*

* *


**Brillouin scattering (BS) spectroscopy.** BS represents an inelastic light scattering technique, similar to the Raman spectroscopy. However, the energies of the excitations probed in the BS spectroscopy are much lower than those probed in the Raman spectroscopy and they lie well below 50 $cm^{-1}$. The excitations that can be investigated with BS spectroscopy include acoustic phonons, magnons, spin waves, surface ripples, diffusive excitations, etc. Acoustic phonons. produce strain, causing the change of mass density, and modulate the dielectric constant of the material. The BS spectroscopy allows for the monitoring of elastooptic properties of the material, the determination of sound velocities and elastic constants, the investigation of the effects such as anharmonicity, the phase transitions. It also provides information on the interaction of acoustic phonons with different excitations, electronic states, polaritons, and plasmons. The spectra are usually recorded in the backscattering geometry since forward scattering can be performed only on transparent samples.

Since the energy of the scattered radiation is very close to the initial, laser line energy, differing from it often for less than an inverse centimeter, a grating monochromator cannot be used as a spectrograph in this experiment. Instead, a Fabry Perot interferometer is used (plane or spherical). The plane Fabry Perot interferometer consists of two parallel disc-shaped mirrors made of glass or quartz mounted at a constant distance $L$ one from the other. The flat mirror surfaces are coated with such a material that most of the light incident upon them is reflected and only a small fraction is transmitted. The interference of light after numerous reflections has maxima for wavelengths: $\lambda = 2L/p$ ($p$ is a non-zero integer) and by transmitting mostly light of these wavelengths, the individual wavelengths are separated from the rest of the scattered radiation.

Our Brillouin scattering system, the Tandem Fabry Perot Interferometer TF-1 (JRS Scientific), consists of two interferometers placed with their main axes being almost parallel (inset of Fig.14), with a small offset angle $\varphi$ that their mirror distances are almost the same, having values $L$ and $L\cos\varphi$. The light is passed three times through each interferometer (six passes in total) and improves the contrast drastically. Two interferometers are piezoelectrically scanned and their work is synchronized by mounting their scanning mirrors on the common scanning stage. Also, by placing the interferometers in proximity to each other the system is less sensitive to temperature fluctuations. The mirror spacing can be set in the range (0-50) mm, whereas small mirror movements with scanning amplitude of up to 2μm are made possible by use of deformable parallelogram beneath the mirrors holder and an attached transducer controlled electronically. Both

interferometers are isolated from the low-frequency vibrations (such as building vibrations) with a vibration free system AVI 350 LP.

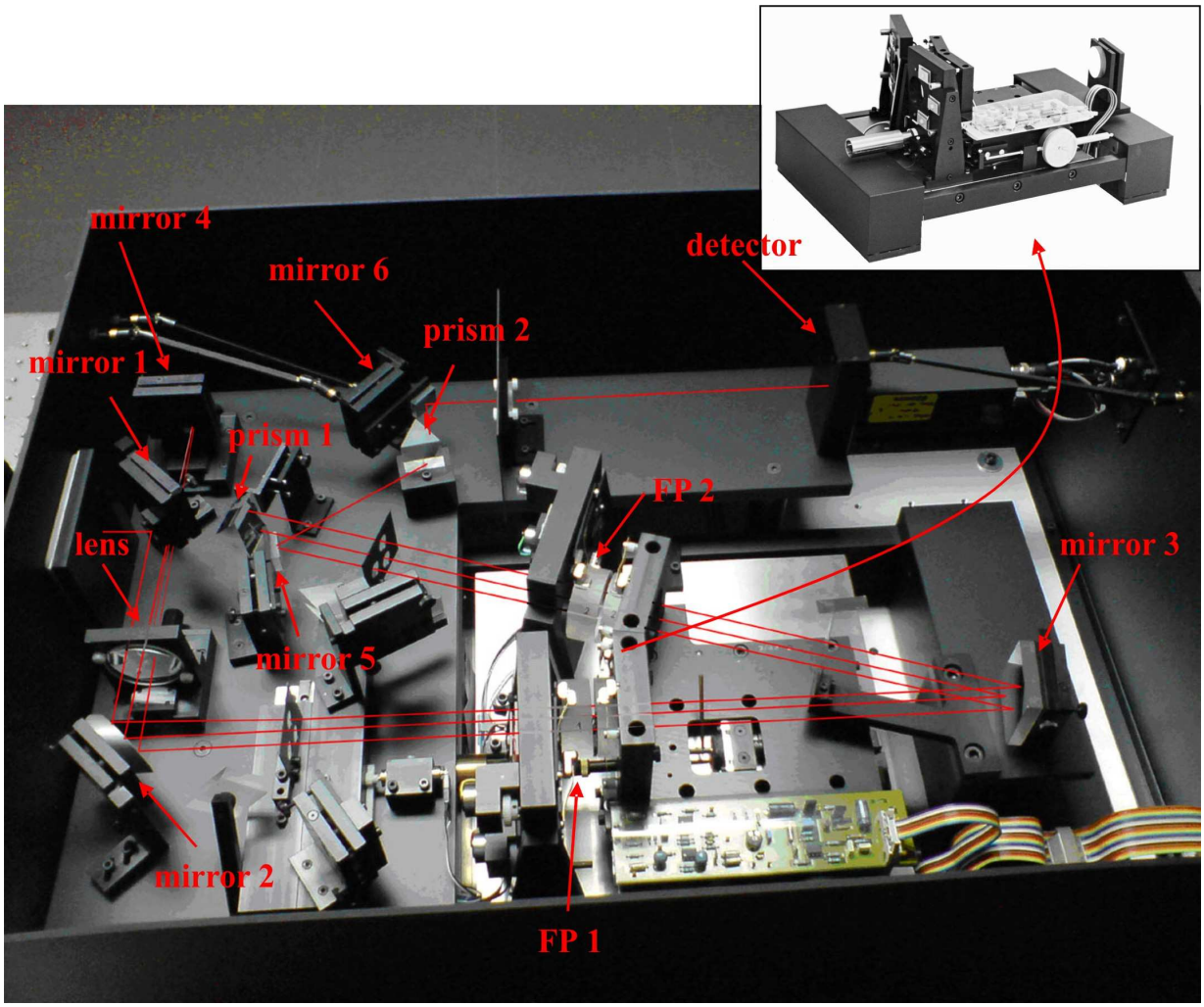



*Figure 14. Brillouin JRS Scientific spectrometer. Inset: Tandem Fabry Perot interferometer with a common scanning stage*

A complete spectrometer is shown in the Fig. 14. On the left side the beam enters the system through the aperture, then is reflected from the small mirror 1, collected and collimated with the lens, reflected from the mirror 2 and after this, it enters the Fabry Perot interferometer 1 (FP1). After a signal intensity amplification in the FP1, the beam is reflected from the mirror 3, passes through the second interferometer (FP2), is refracted and reflected from the prism 1 and returns through the FP2 and FP1 to the lens and is focused on the very small mirror 4 (cat's eye) behind the first mirror. From the mirror 4, the beam returns again through the FP1 and FP2, but now is reflected from the mirror 5, refracted through the prism 2, reflected from the mirror 6 and through a small lens is directed to the detector. It is clear that the beam passes three times through a FP1 and three times through a FP2. The distance between the mirrors is set by using a control on the front board that runs a motor that moves the translation stage. The adjustment of the FP1 and FP2 mirrors positions is performed also with x- and y-position controls on the front board, with a purpose of making FP1 and FP2 transmit. A fine adjustment of these positions is performed with the help of corresponding controls on the control unit. The beam transmission is controlled visually, checking

that the entire beam spot is viewed on a piece of paper placed after the FP1 and the FP2, after mirror 6 etc. The described procedure relates to the tandem operating mode of the spectrometer. There is one more working mode, the alignment mode, with a slightly changed geometry, which is characterized by the collection of the light reflected from the FP1 and the FP2, which allows for viewing dips at the positions in the spectra corresponding to the wavelengths transmitted through the interferometers. This operation mode makes it easier to do final adjustments of mirror positions in order to ensure that the interferometers transmit completely. After this a measurement is made by setting the scanning amplitude on the control unit and collecting the spectra. The signal from the detector (photon counting module Perkin Elmer MP 983) is analyzed with a MCA Ghost multichannel analyzer.

Detector incorporated in the experiment is PerkinElmer Photon Counting Module MP 983, with spectral sensitivity in the region (185-650) nm. Photocatode used is a low noise bialkali, with dark counts of 2cps, and maximum counting rate of 10MHz. Detector and the spectral response of the photocatode (curve signed C983) is shown in the figure 15.

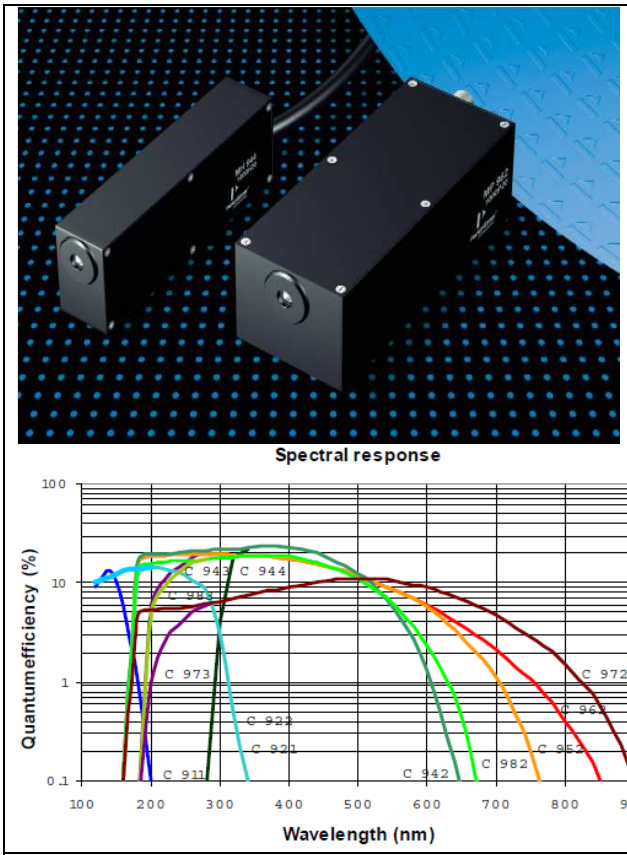



*Fig. 15. Photon Counting Module MP 983, with spectral sensitivity –curve C983.*


*Fig. 16. Coherent Compass 315 M laser*

Excitation source in the BS spectroscopy experiment is a Coherent diode-pumped Nd:YAG laser, model Compass 315M. It is a single frequency, single mode laser operating at the wavelength of 532 nm with maximum power of 150mW, Fig. 16..

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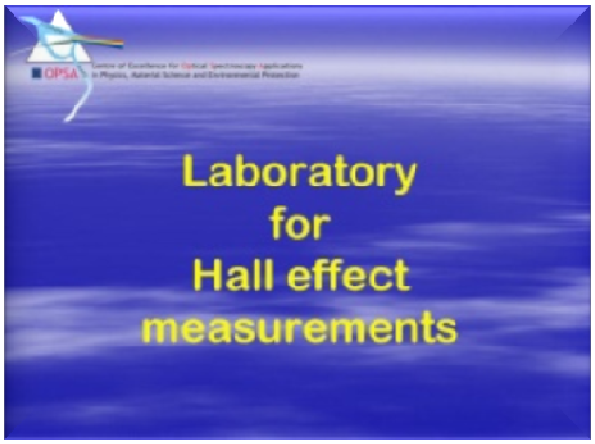
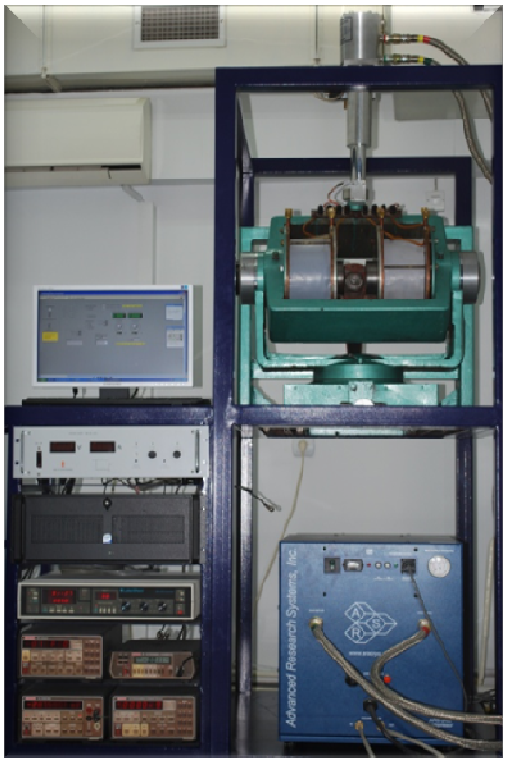



**Laboratory for transport properties measurements (Hall effect set-up)** is equipped with a conventional electro-magnet (magnetic field up to 1.5 T), a Hall effect set-up and an ARS Displex DE-202N closed-cycle-helium cryostat (for low temperature measurements between 7 and 300 K).

**The transport properties measurements (Hall effect experiment) consists of:**

- **The Electromagnet (**hand made**).** This electromagnet can realize a magnetic field of up to 1.5 T for pole distance of 1 cm. In case of low temperature measurements the pole distance is 7 cm and the maximum value ofthe magntic field is 0.5 T.
- **The Current source** for electromagnet is Delta electronika series 3000, model SMD: 120V-25A.
- **The Keithley 220 Programmable Current Source** – (DC current range: 2nA – 100 mA).
- **The Keithley 705 Scanner** with **Keithley 7065 Hall Effect Card,** which is designed to assist in making resistivity and Hall voltage measurements on several types of semiconductor speciments.
- **The Keithley 196 System DMM** – is a five function autoranging digital multimeter. The DC voltage measurements from 100 nV to 300 V. Resistance measurements from 100 μΩ to 300 M Ω. AC voltage measurements from 1 μV to 300 V. DC current measurement from 1 nA to 3 A., and AC current measurements from 1 nA to 3A.
- **The Close cycle helium gas cryostat** (ARS, model Displex DE-202N) for temperature measurements




*Hall effect measuring system.*

between 6 and 300 K..

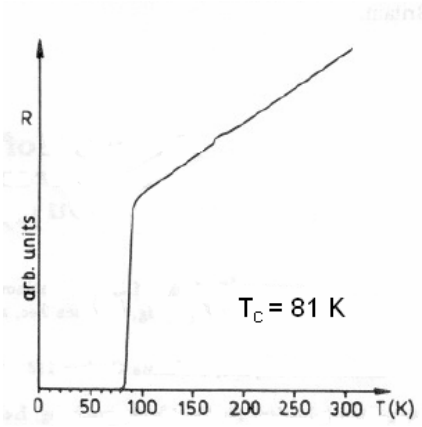
- **The Lake Shore DRC91CA Temperature Controller**.
- **A Pfeiffer Vacuum pump** for air evacuation from cryostat.
- **A PC computer** for experiment running and data collecting.

**Measurements:**

Using the Hall-effect set up it is possible to measure:

1. Specific electrical resistivity using the Van der Pauw method.

2. The Hall voltage, i.e. the Hall constant. Combying this and the electrical resistivity data it is possible to determine carriers type, concentration and mobility.




*Electrical resistivity of $YBa_2Cu_3O_7$ superconducting oxide.*

Specific electrical resistivity measurements are done using the Van der Pauw method. At first, it is necessary to create 4 electrical contacts on the sample. The electrical resistivity is measured by applying the current between two adjacent terminals and reading the voltage at the other two terminals. The total of 8 measurements is made for all possible combinations of measurements among the 4 contacts. A specific electrical resistivity is calculated according to the Ohms law using values of applied currents and measured voltages, taking into account the proportionality coefficients based on the shape of the sample.

*

* *


**Laboratory for magnetic and magneto-optic measurements** is equipped with a 14 T cryogen free measurement system (Cryogenic Ltd. superconducting magnet with a vibrating sample magnetometer, a resistivity and Hall effect set-up, a specific heat system). This system has an optical window at the bottom of the cryostat for optical connection with the TriVista TR557 triple

Raman system so that Raman and photoluminescence measurements can be performed at low temperatures and in high magnetic fields.

## High Magnetic Field Measurement System

The High Magnetic Field Measurement System (HMFMS) made by Cryogenic is a very versatile system for various experiments and measurements in high magnetic fields up to 14 T, within the temperature range from 1.6 K to 325 K. The HMFMS is capable of measuring several physical quantities:

- Magnetic moment and magnetization using a Vibrating Sample Magnetometer (VSM)
- AC Susceptibility
- Electrical resistivity
- Hall Effect constant
- Specific Heat

The HMFMS is a “cryogen free system”, meaning it does not use liquid helium for cooling superconducting magnets. Instead, it uses a closed cycle system of helium gas cooling with a compresor. The main advantage of a cryogen free system is the low operating cost. No liquid helium or nitrogen is required for cooling down or operation; there are no costs associated with storage and transport of liquids. Safety issues and training for personnel are minimised. It is also simple to use: switch on the cooler and wait for it to reach the operating temperature; the system is then ready to use.

In the core of the system is a superconducting magnet, based on a NbTi/$Nb_3Sn$ alloy. The magnet is able to achive magnetic fields of up to 14 T, with a magnet energation rate up to 0.7 T/min, and with a central field homogeneity of 0.001% over 25mm. The sample is mounted on a probe which is placed inside the Variable Temperature Insert (VTI) which allows for sample temperature variation in the 1.6-325K range. The temperature stability is ± 0.05 K, and a typical cooldown time for the entire system is about 28 hours.

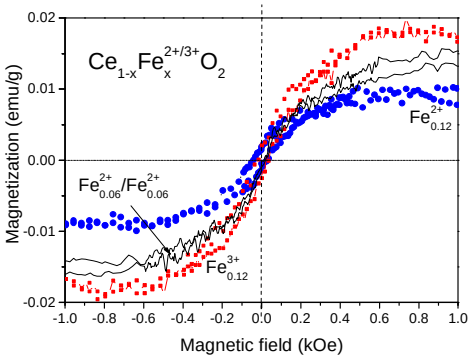
By changing a probe holder it can be easily changed the type of measurement performed on the system, i.e. the system can be used in different configurations for different experiments. The system is using a LabView based software for monitoring and data collection which enables automated multisequence measurements.

## Vibrating sample magnetometer

The Cryogenic VSM is a research instrument for measurements of the DC magnetic moment in the fields of up to 14 Tesla. In order to generate a signal proportional to the magnetic moment, the sample is set to vibrate in a constant (or slowly varying) applied field. The signal is detected by an astatic pair of pick-up coils. The coils sense the variation of magnetic flux due to the sample movement. The typical frequency of vibration is around 20 Hz, and the amplitude is 1 mm. The

pick-up coils are located inside the VTI within the bore of the superconducting magnet. The signal is detected by a lock-in amplifier.

**Specification**
Vibration frequency: 1 – 100 Hz, typically 21 Hz
Maximum sample size: 10 mm
Optimum sample size: 5mm or smaller
Noise base (10 s averaging) : $10^{-6}$ emu
Dynamic range (standard): $10^{8}$
Accuracy and reproducibility: 0.5%




*Magnetization of $Ce_{1-x}Fe_xO_2$ samples (right).*

## AC susceptometer

The AC magnetic susceptibility of a sample is measured by applying a small AC field using a drive (or primary) field coil and detecting the sample response with an astatic pair of pick-up (or secondary) coils.

In the absence of the sample the coils are balanced so that the output signal is zero. When the sample is positioned inside one of the pick-up coils, the balance is disturbed, and the resulting output signal is proportional to the sample susceptibility. Generally, there is a phase shift between the AC field and the sample response. Therefore the dynamic susceptibility is usually represented in terms of the real and imaginary parts. The real part is the characteristic of reversible processes, and the imaginary part characterises the energy dissipation.

**Specification**:
Maximum AC field amplitude: 20 gauss at 100 Hz
Sensitivity: at 1 KHz $10^{-7}$ emu at 4K
Useful frequency range: 1 Hz – 20 KHz
Maximum sample diameter: 5 mm
Sample temperature range (VTI): 1.6 – 325 K

## Specific heat measurements

The Cryogenic Ltd. miniature AC calorimeter is designed to measure the heat capacity of samples weighing as little as one microgram.

The AC calorimeter is based on an AC technique that offers unsurpassed sensitivity combined with simplicity of operation. The sample is mounted onto the calorimeter membrane using thermally conductive vacuum grease mounted in a closed cell with low-pressure exchange gas. The complete probe is loaded through an airlock into the VTI.

**Specification**:
Temperature range: 5 – 300 K
Frequency of temp. modulation: 0.1 – 100 Hz
Amplitude of temp. modulation: 0.01 – 0.1 K
Typical sample dimensions: 100 x 200 microns
Typical sample mass: 1 -10 μg.

## Electrical resistivity and Hall effect measurements

The Resistivity system is configured for 4-terminal resistance measurements and Hall voltage measurements in the range from μΩ to MΩ resistance. The resistance is measured by a high quality digital voltmeter and a current source. The standard sample platform is typically 15 mm x 50 mm with 8 electrical connections for two samples.

*Probe for Electrical resistivity and Hall-effect measurements*

**Specification**:
DC resistance measurement range: $10^{-7}$ to $10^{-8}$ Ω
DC voltage sensitivity: typically 0.1 μV to 1 V
Accuracy: Higher than 0.1% for 1 – $10^{-6}$ Ω
Resistance frequency range: Normally DC
Sample size: up to 10 mm

# *Human resources*

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Director of Center for Solid Sate Physics and New Materials

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**Research interests:**
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**Research interests:** Raman, infrared and photoluminescence studies of nanostructured materials. Numerical simulations of the vibrational properties of nanomaterials.

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**Research interests:** FTIR, Raman and photoluminescence spectroscopy of nanometric non-oxide ceramics, nano composites and semiconducting nanopowders and thin films. Magnetic properties of nanomaterials.

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**Research interests:** - Vibrational spectroscopy of lowdimensional systems (polymers, nanotubes,...); Infrared, Raman and photo-luminescence spectroscopy of semiconductor nanoparticles in different matrices.

**Dr Maja Romčević, associate research professor**

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**Research interests**
Vibrational (Raman and IR) spectroscopy and transport measurements of semiconductors and semimagnetic semiconductors - bulk, thin films, and nanoparticles

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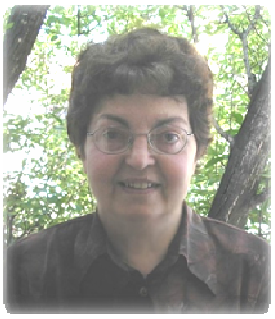

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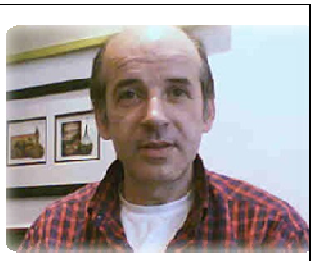

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