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

Report

Project no. **INCO-CT-2006-026283-OPSA**

Project title:

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

Appendix B:

Report from the training courses for μ - Raman and μ - PL measurements at TriVista spectrometer (WP3) and for infrared reflectivity measurements at Bomem DA8 spectrometer(WP4).

Table of contents

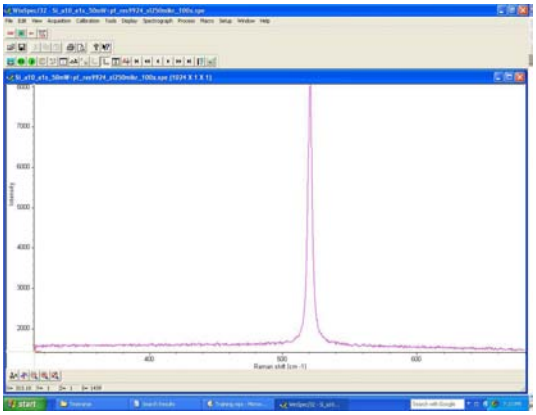
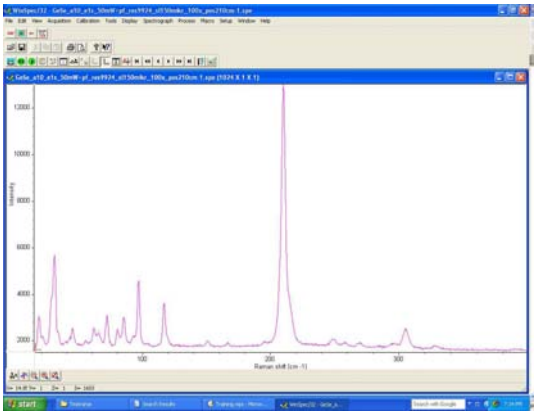
A. Report from the first training course for μ - Raman and μ - PL measurements at TriVista spectrometer (WP3)

Training course for μ - Raman and μ - PL measurements at TriVista spectrometer system was organized in May of 2009. The participants were young PhD students from the Center for Solid State Physics and New Materials.


Photos from training course for μ - Raman and μ - PL measurements at TriVista system

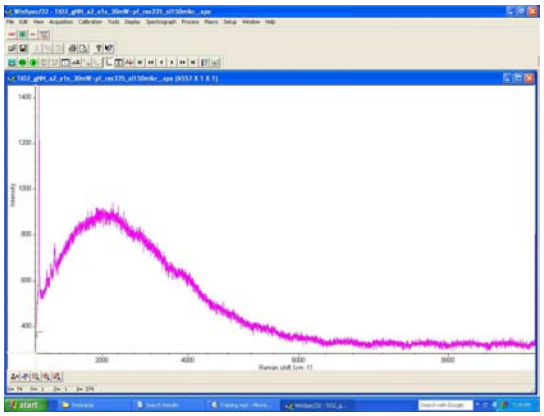
During **the first day** of training the attendants were introduced to the main parts, structure and software that control particular functions of the TriVista 557 system. They were taughed to choose the system settings and to start the measurements according to the Programme of the training course.

During **the second day** of training the attendants have measured the Raman spectra of reference samples (crystalline Si and $GeSe_2$) at room temperature. Some of the spectra obtained during this part of the course are shown in the figure bellow.

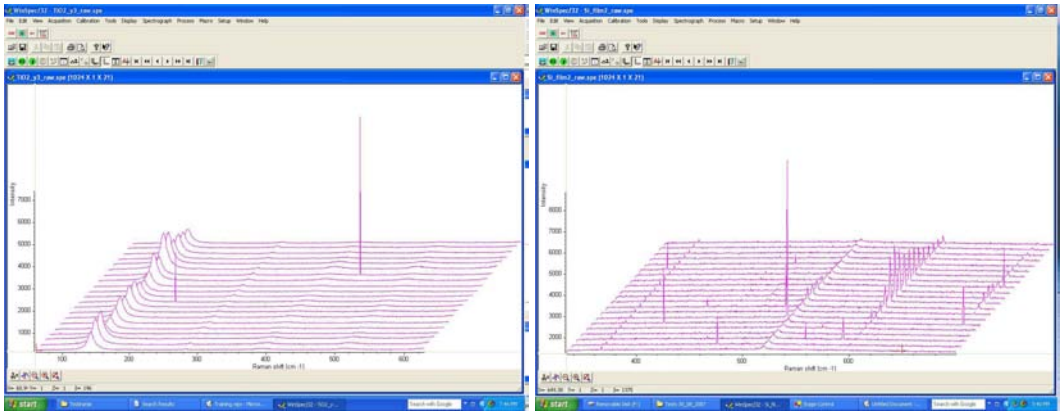
Raman spectra of Si (left) and $GeSe_2$ (right) with magnification of 100x obtained on Tri Vista System using gratings with 900 grooves/mm in first two stage and gratings with 2400 grooves/mm in the last stage

According to the Programme of the training course, the attendants were trained to use some additional features of TriVista system during **the third day**. They have collected the Raman and PL spectra in wider range, using the Step&Glue mod. One of obtained PL spectra of TiO_2 nanopowder, slightly pressed into pellet, is shown in figure bellow.



PL spectrum of TiO_2 nanopowder, excited by 514.5 nm of mixed Ar^+/Kr^+ laser, measured in wide range – up to 9000 cm^{-1}.

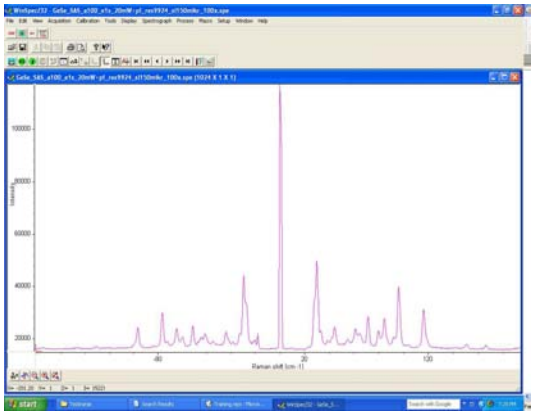
The attendants also have been instructed how to use x, y and z scan function using the Stage Control software for the control of XYZ motorized stage. Some of the Raman spectra obtained using 514.5 nm laser line are shown in the figures bellow.

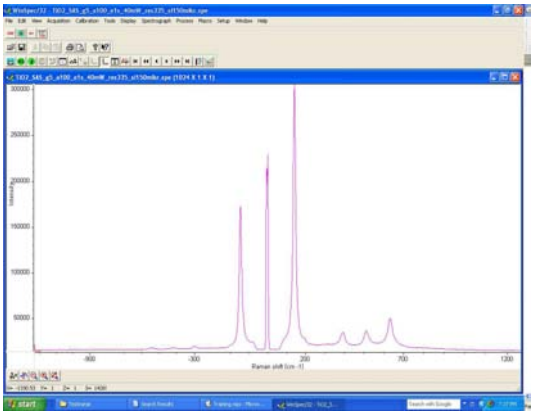


Y-scan: Raman spectra of the TiO_2 at various positions on the sample along y-axis (left); Z-scan: Raman spectra of the Si thin film at various positions on the sample along z-axis (right). Note that intense plasma of 514.5 nm line of Ar^+/Kr^+ laser is visible in the spectra of Si, as they were collected without plasma filter.

TriVista system has the possibility of determining the position of the sample on the z-axis (vertical axis) where the beam is focused on the sample. The autofocus process was demonstrated at the TiO_2 sample.

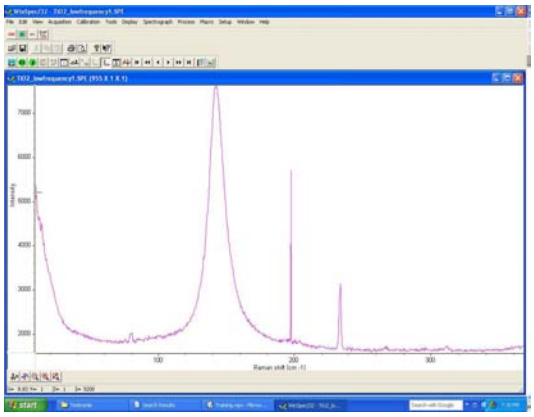
The attendants also have been trained for simultaneous measurement of Stokes and anti-Stokes Raman signal, which is one of the special advantages of TriVista system. Using the laser stop mask with 600μm thick bar as a laser line blocker in the middle of the intermediate slit, the Raman spectra of $GeSe_2$ reference sample and TiO_2 nanopowder were obtained (figure bellow).





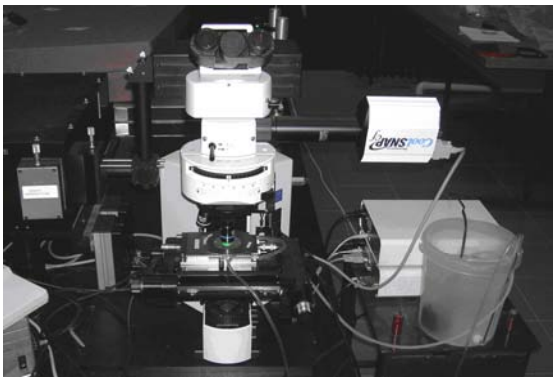
Stokes and anti-Stokes Raman spectra of $GeSe_2$ with 900/900/2400 gratings configuration (left) and TiO_2 with 300/300/500 gratings configuration (right)

Finally, Raman measurements in the spectral region very close to the laser line are demonstrated on TriVista system, as can be seen from low frequency Raman spectra of TiO_2 nanopowder in the figure bellow.



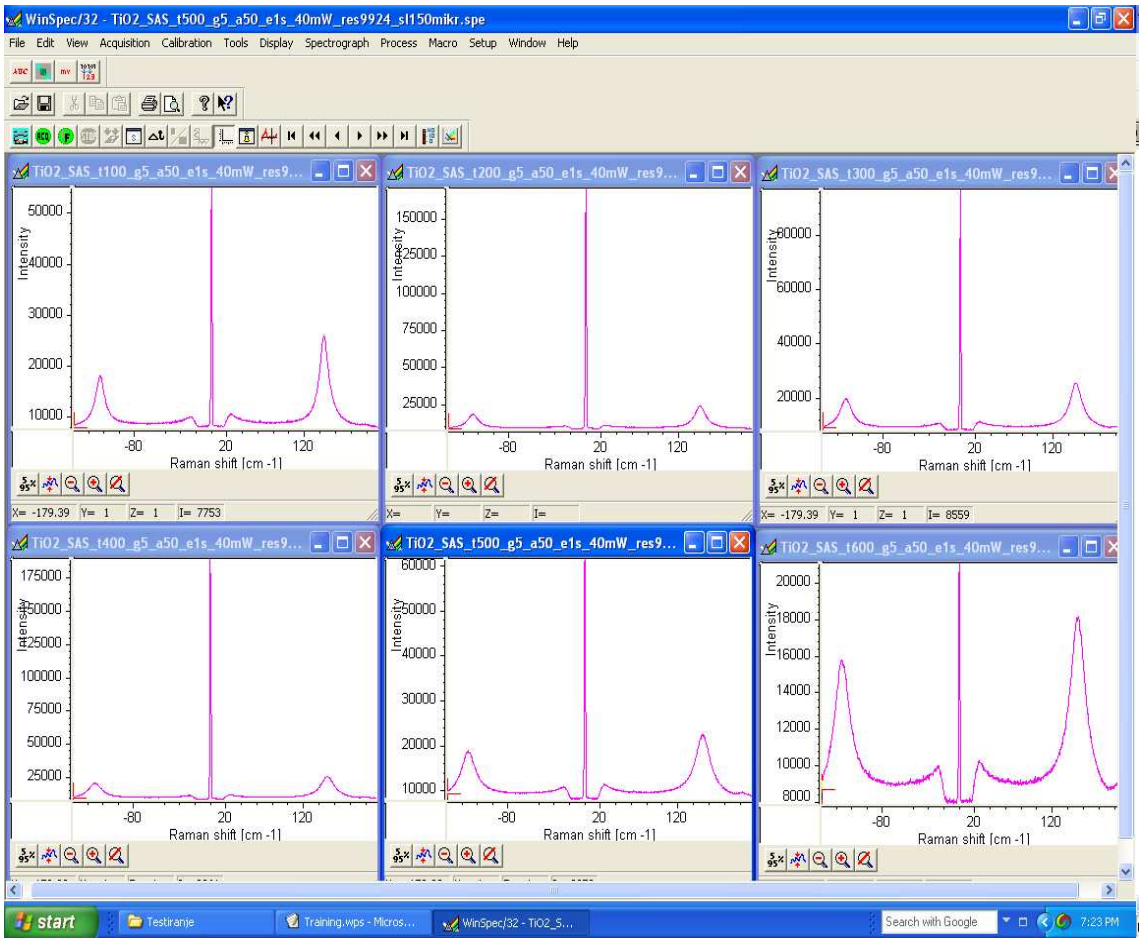
Raman spectra of TiO_2 with output laser power of 20 mW and 900/900/2400 gratings configuration.

Forth day of training was dedicated to the variable temperature Raman measurements using Linkam THMSG 600 heating/cooling stage. The attendants of the course have been trained to mount Linkam microchamber at XYZ stage (figure bellow) and to use temperature controller and liquid nitrogen pump.



Heating/Cooling Linkam Stage mounted at TriVista system.

After demonstration of heating and cooling procedure, the Linkam software which controls the heating and cooling in Linkam microchamber was started. The Raman spectra were collected simultaneously in Stokes and anti-Stokes region at specified temperature. The spectra obtained from sol-gel synthesized TiO_2 nanopowder in the temperature range 100-600^oC are shown in figure bellow.



Stokes and anti-Stokes Raman spectra of TiO_2 nanopowder measured at 100, 200, 300, 400, 500 and 600^oC shown from upper left to lower right corner of the figure, respectively. The 900/900/2400 gratings configuration were performed.

The last day of the course was dedicated to optical path adjustment of the TriVista spectrometer, after the replacement of mixed Ar^+/Kr^+ laser by HeCd laser. First of all, the alignment the laser beam path outside the TriVista system (from the laser to the entrance of TriVista) was done. Afterwards, the alignment of the beam path inside the confocal micro-Raman interface (microchamber at the figure bellow) and at the entrance of the first stage of TriVista spectrometer was performed. Note that alignment procedure is time consuming process which can take few hours.

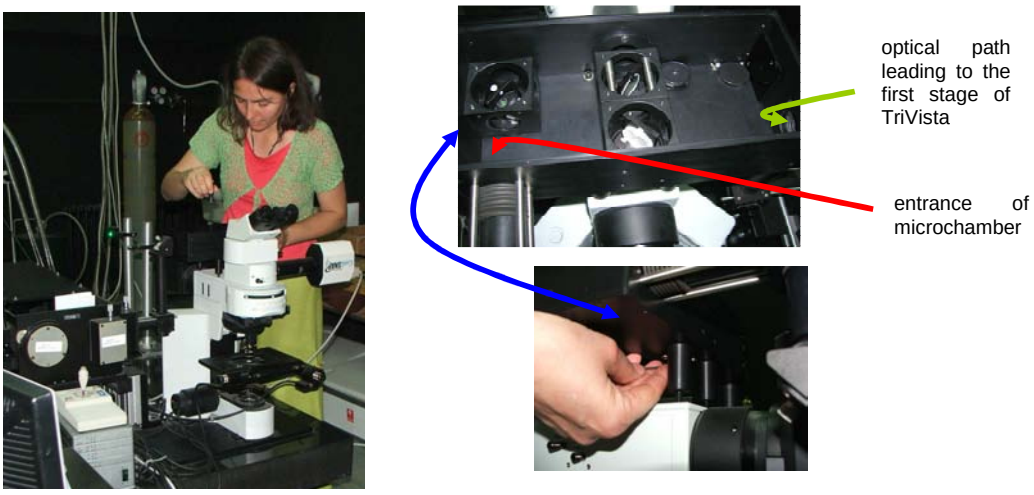
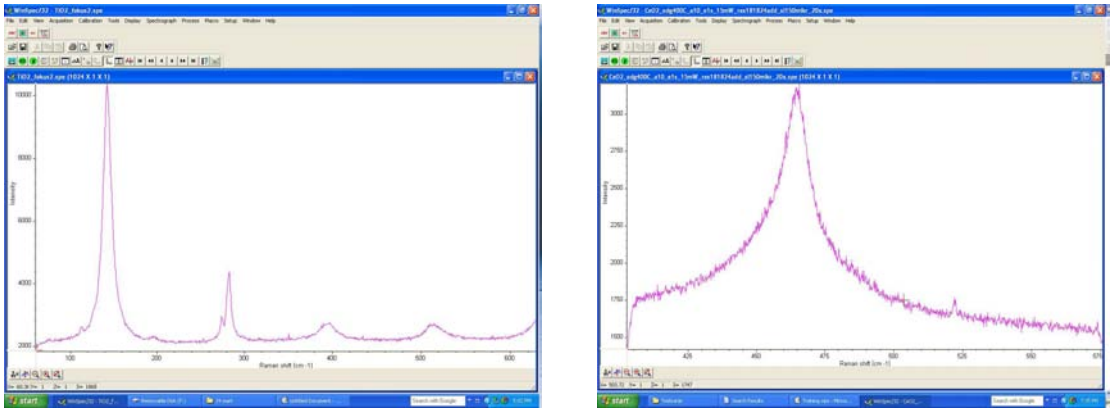


Illustration of the beam path alignment inside the confocal micro-Raman interface (microchamber)

Optical path adjustment always must be followed by matching the stages in TriVista spectrometer. After this software procedure TriVista Raman spectrometer was again ready for measurements. The Raman spectra of TiO_2 and CeO_2 nanopowders measured using 441.6 nm line of HeCd laser are shown in the figure bellow.



Raman spectra of TiO_2 and CeO_2 nanopowders. In TiO_2 spectrum tri plasma lines of 441.6 nm line of HeCd laser at about 113, 274 and 184 cm^{-1} are visible.

This five days training course has prepared researchers for substantive μ - Raman and μ - PL measurements at TriVista spectrometer. The attendants have demonstrated skills for Raman measurements and obtained spectra of good resolution and high intensity both from reference samples (Si and $GeSe_2$) and investigated nanomaterials (TiO_2 and CeO_2 nanopowders). They have also shown their efficiency in using the additional possibilities of TriVista system: Raman and PL measurements in extended range, simultaneous Stokes/anti-Stokes measurements, low-frequency Raman measurements, operation in X-, Y-, Z-scan mode, autofocus function, as well as variable temperature Raman measurements.

B. Report from the training courses for infrared reflectivity measurements at Bomem DA8 spectrometer (WP4)

The course of infrared reflectivity measurements is dedicated to the PhD students and other attendants as an introduction in using infrared spectroscopy for characterizing, identifying or determining a substance. The participants were instructed about design of spectrometers, about the sample preparation, diverse methods of measurements, modelling and interpretation of the spectra. The course was dedicated presumably to the application of IR spectroscopy to nano-sized systems. It was organized into 10 lectures. Duration of course was two weeks. The titles of lectures were:

- **Introduction to the spectroscopy, classification of methods and common types of spectroscopy**
- **Background and theory of infrared spectroscopy**
- **Infrared instrumentation**
- **Fourier transform infrared spectroscopy**
- **Dielectric properties: classical treatment**
- **Determination of ε (ω) from experimental data**
- **Standard accessories for transmission and reflectivity measurements**
- **Reflectivity measurements**
- **Transmission technique**
- **Introduction to infrared spectroscopy characterization of nanostructured systems**

All our PhD students from the Center for Solid State Physics and New Materials past this course.

PhD student and teacher after finishing IR course.