



DLF

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UNIwersytet Gdański

Experiment 22

Raman spectra of single crystals of silicon (Si) and diamond (C)

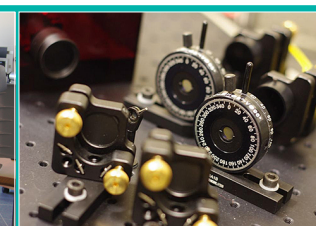


KAPITAŁ LUDZKI
NARODOWA STRATEGIA SPÓJNOŚCI



UNIwersytet Gdański

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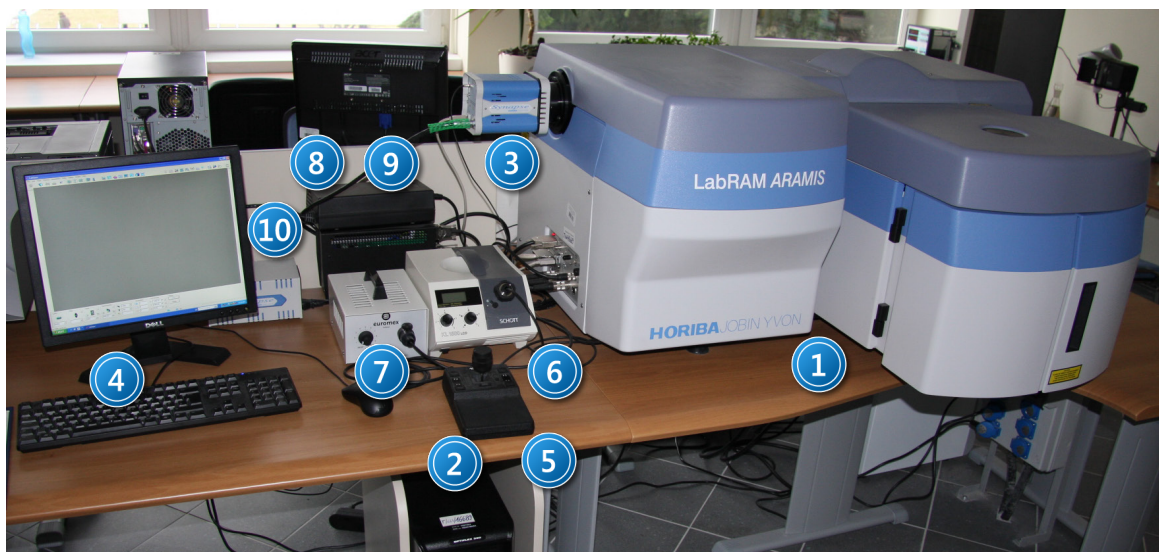


I. Background theory.

1. Motion of nuclei in diatomic and polyatomic molecules:
 - a) harmonic oscillator model;
 - b) anharmonic oscillator model.
2. Molecular vibrations of polyatomic molecules.
3. Oscillating spectra of polyatomic molecules.
4. Lattice vibrations, optical and acoustic phonons.
5. Electron structure of molecules:
 - a) electron states in molecules;
 - b) energies of electron states;
 - c) molecular orbitals.
6. Origin of energy bands in solids:
7. Crystal structure of diamond and silicon:
 - a) covalent bonds;
 - b) crystal symmetries.
8. Crystal structure of graphite and fullerenes.
9. Types of bonds in carbon atoms.
10. Electronic band spectra of molecules:
 - a) absorption of radiation;
 - b) Franck-Condon principle;
 - c) transition moment;
 - d) selection rules for optical transitions;
 - e) emission of radiation;
 - f) infrared absorption spectrum.
11. Classical theory of Raman scattering:
 - a) Rayleigh scattering;
 - b) Raman resonance scattering;
 - c) Raman scattering spectrum – Stokes and anti-Stokes spectrum;
 - d) polarisation of scattered radiation.
12. Experimental setups to study Raman spectra:
 - a) monochromator – optical system and principle of operation;
 - b) design and operation of lasers;
 - c) photomultiplier tube – construction and use.

II. Experimental tasks.

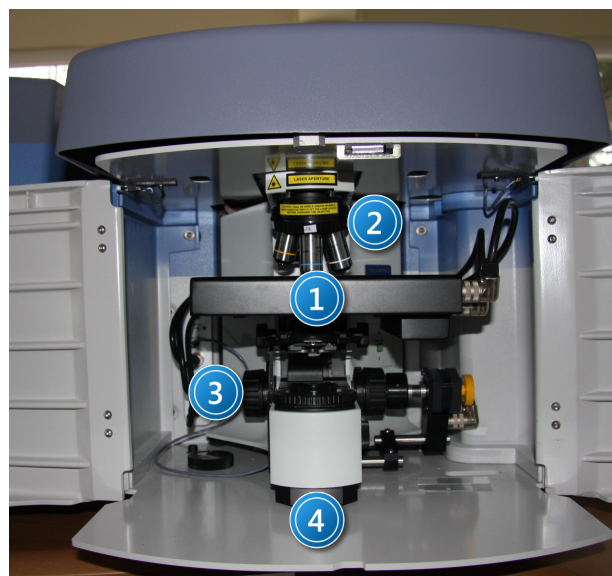
1. Refer to the setup shown in *Pictures 1, 2 and 3.*



Picture 1. Measurement apparatus – front view : 1 – Raman spectrometer ; 2 – computer; 3 – CCD camera; 4 – monitor and keyboard; 5 – control joystick; 6 – microscope illumination; 7 – microscope illumination for transparent samples; 8 – transformer; 9 – power to sample mounting table; 10 – CCD camera power supply.



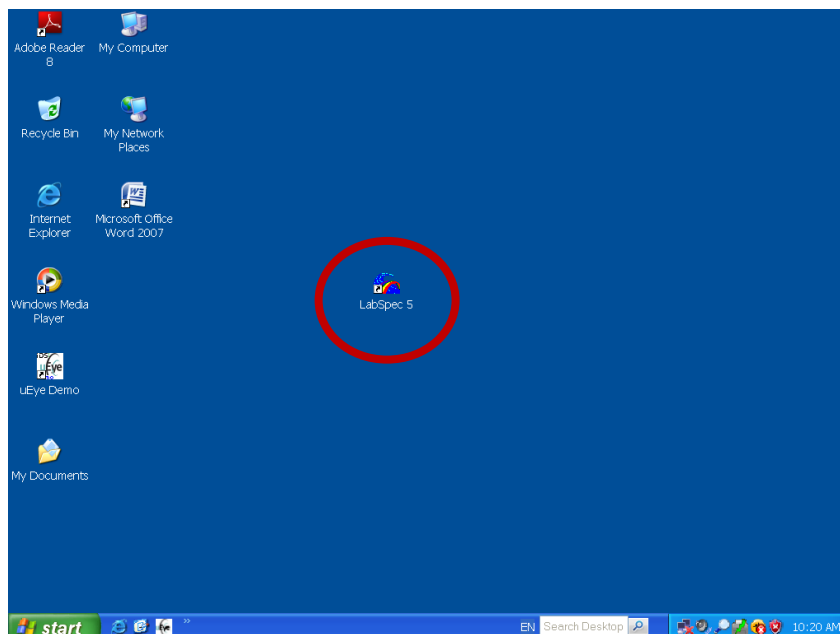
*Picture 2. Measurement apparatus – side view:
1 – He-Ne laser power supply; 2 – YAG:Nd laser power supply; 3 – Raman spectrometer.*



Picture 3. Spectrometer chamber: 1 – moveable x, y, z sample mounting table; 2 – objective lenses; 3 – z-axis adjustment screw; 4 – condenser with polariser.

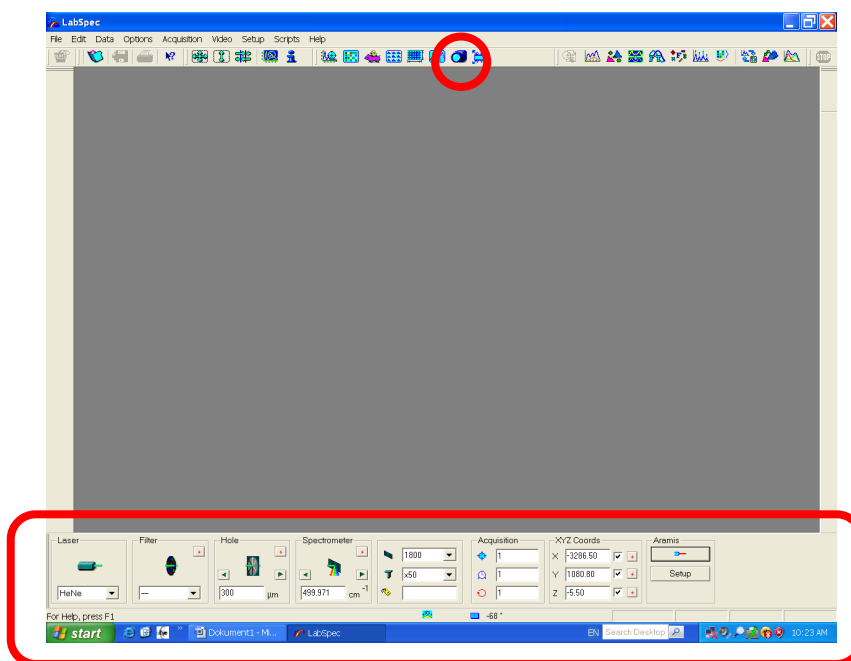
2. Turn on the power for the Raman spectrometer, CCD camera and the xyz confocal microscope table using the power strip switch.

3. Turn on the He-Ne laser by turning the laser power ignition key in the housing (1 in Picture 2) (a green light will glow on the housing).
4. Turn on the computer. The Windows XP desktop appears on the screen. Start the software "LabSpec 5" (the marked icon in Picture 4).



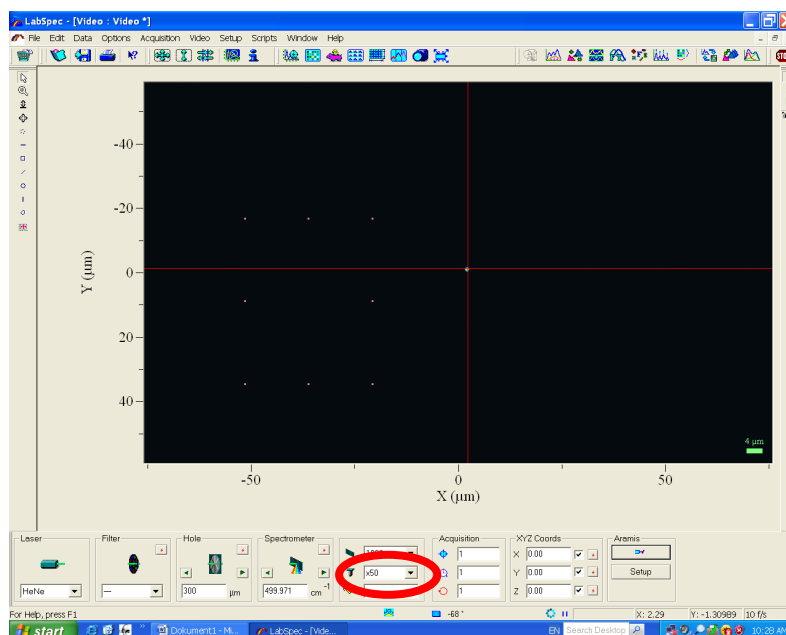
Picture 4. View of the screen before starting the measurements. The program for measuring Raman spectra is circled.

You will see a window as in Picture 5.



Picture 5. View of the screen indicating video mode selected. The circled video mode icon (at the top) and the measurement settings menu (at the bottom).

- Turn on the camera – (video mode icon is indicated in *Picture 5*).
The video mode screen appears on the screen (*Picture 6*).



Picture 6. Lens selection icon. For S and C select lens x50LWD.

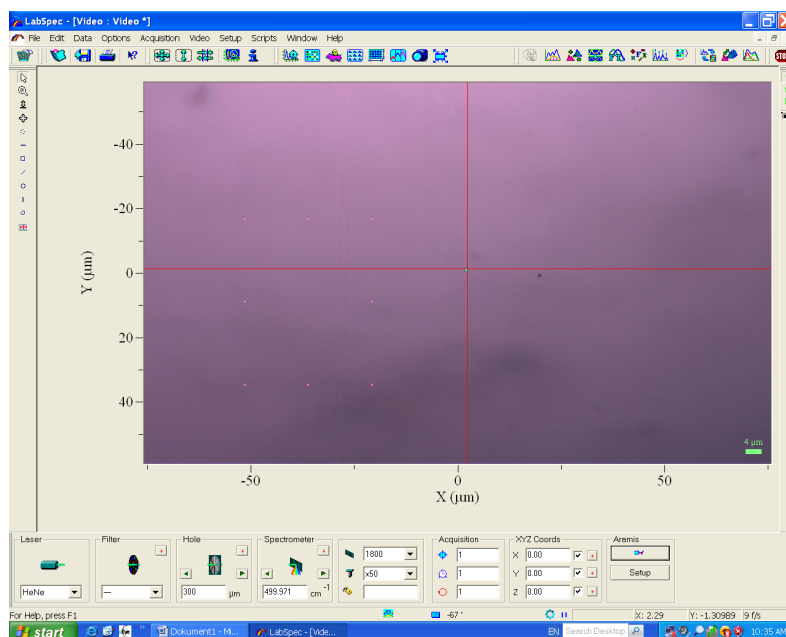
- Select lens x50LWD for measurements in silicon and diamond in the spectrometer chamber (*Picture 3*). Select the same lens in the window “measurement settings” (*Picture 6*).
- Turn on the upper illumination KL 1500 (6 in *Picture 1*). The switch is found in the lower part of the panel. Set the left dial to position 4 and the right dial to position C.
- Place the sample of diamond or silicon on the microscope table. Turn the “z-axis” knob (3 in *Picture 3*) to focus the beam on the sample so that an image of the surface of the sample appears on the monitor (*Picture 7*).
- Focus the image by gently turning the joystick knob left or right. An adequate image of the sample surface is shown in *Picture 8*.

Confirm the current position of the sample table in the software by clicking the three red buttons in the XYZ Coords icon. This will zero the coordinates of the sample table.

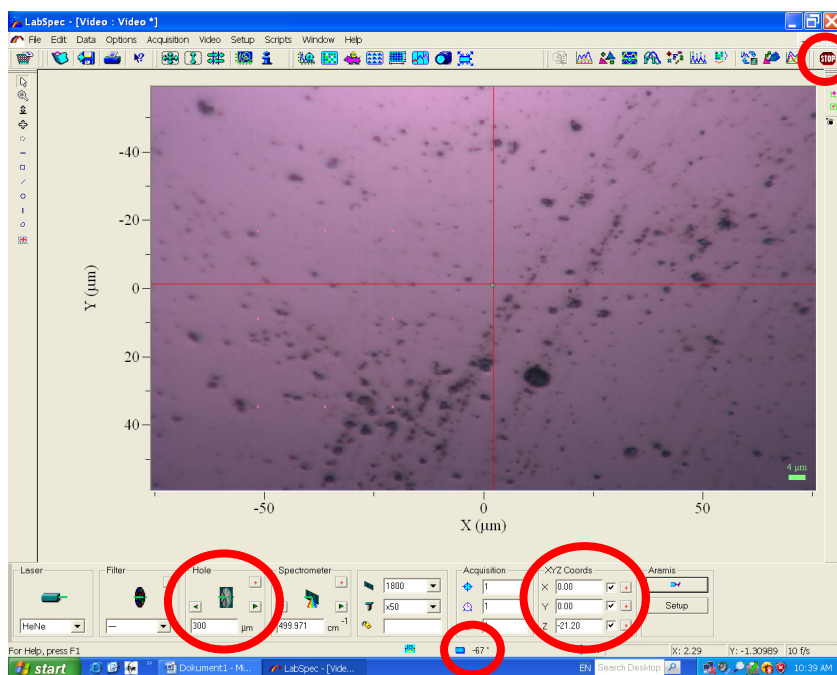


Hint

In case you lose image sharpness, you can always return to the default position by entering zeroes for X, Y and Z in the XYZ Coords window.



Picture 7. Blurred image of the surface of the sample.



Picture 8. A clear picture of the sample surface. Indicated: Stop icon (top); microscope aperture icon (Hole) and the control of the microscope table position (XYZ Coords), temperature detector (bottom).

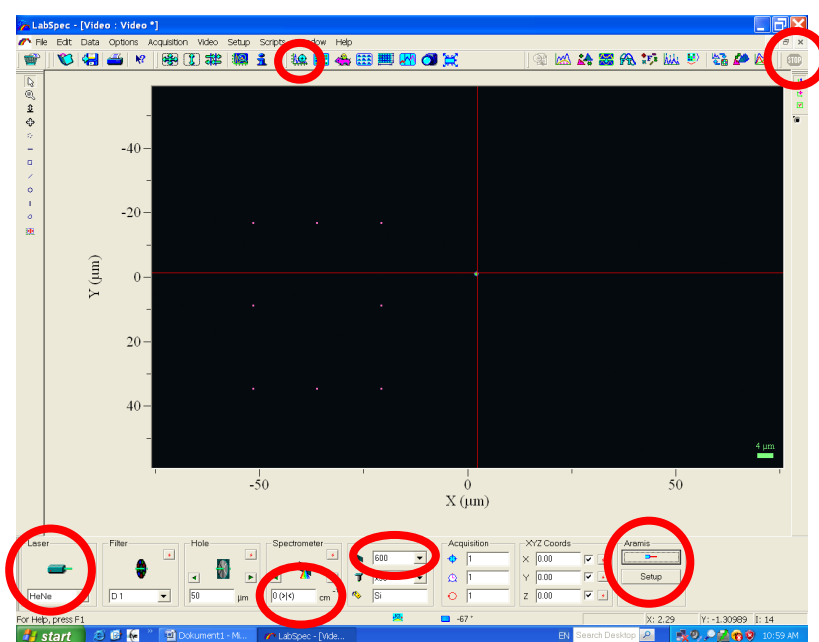
10. Check the temperature of the detector – it should be lower than -60°C (Picture 8).
11. Turn off the upper illuminator (6 in Picture 1) with the switch on the bottom of the panel.
Turn off video mode by clicking the Stop icon (Picture 8). Wait about 10 seconds.



ATTENTION!

It is essential to turn off video mode while measuring the spectrum. Otherwise, you may damage the camera.

12. To record measurements, go to real-time mode (Real Time Domain – RTD). Prior to activating RTD, check the settings in “measurement settings” at the bottom of the screen (Picture 9):
 - Laser – select He-Ne laser;
 - Filter – set to D1;
 - Hole – type 50um and press enter;
 - Grating – choose 600/mm;
 - Lens – choose x50 LWD;
 - Acquisition – type the following parameters in the next window: 1,1,1 or 1,1,10;
 - Ensure that all XYZ Coords in all windows show as 0, 0, 0;
 - Turn on the laser by activating the Aramis window;
 - Spectrometer – enter the value 0 cm^{-1} .



Picture 9. Setting parameters for recording in RTD mode. Indicated: RTD mode icon and Stop (at the top) and laser selection icons, zero-order spectrum, grating, laser on – off (at the bottom).

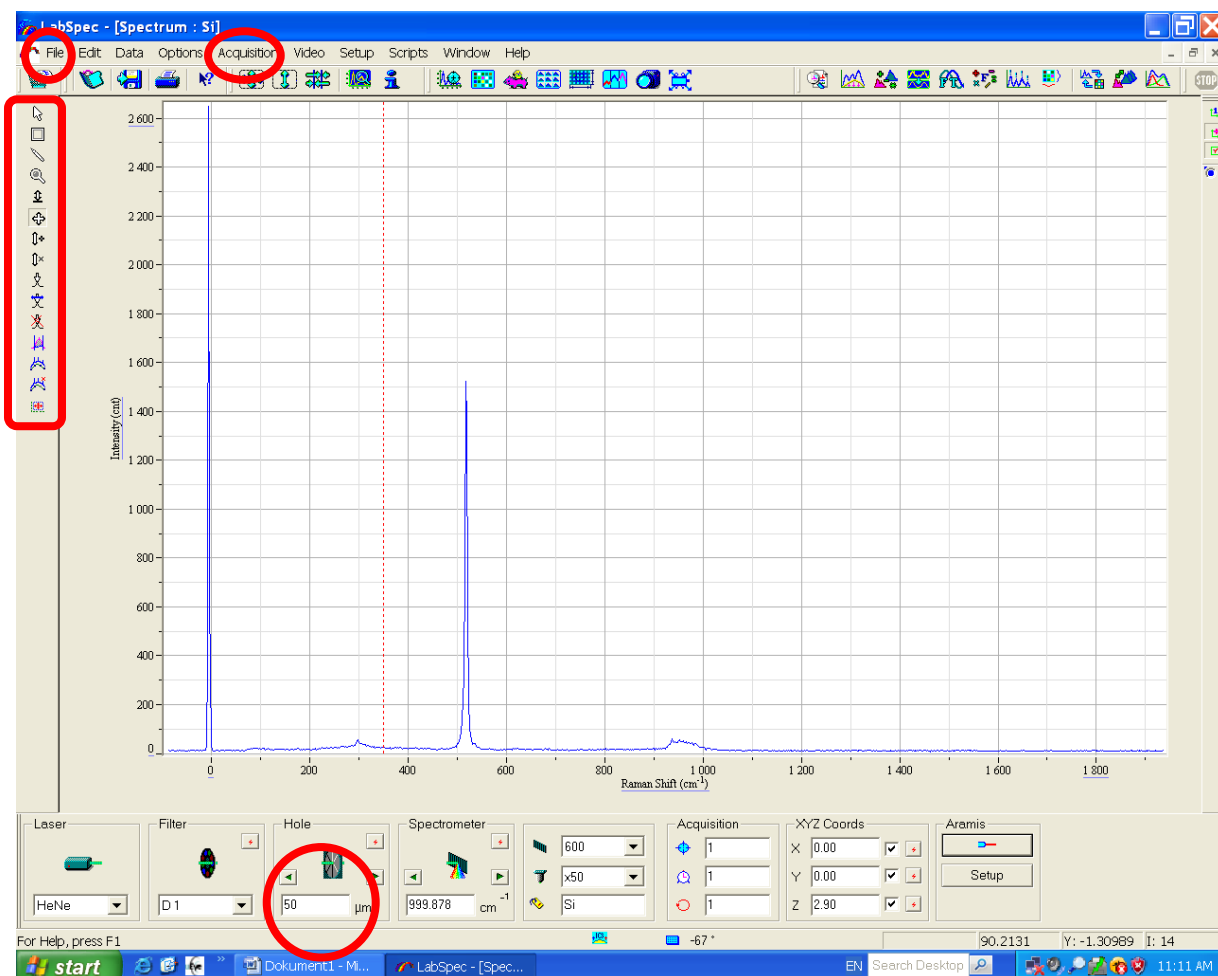
13. Enable RTD mode by clicking the RTD icon at the top of the screen (Picture 9).

If the line displacement on the screen is not larger than 1 nm then proceed to take further measurements.

14. Stop the measurements by clicking Stop, Set a value of 1000 in the spectrometer window and re-enable RTD mode by clicking the RTD icon at the top of the screen.

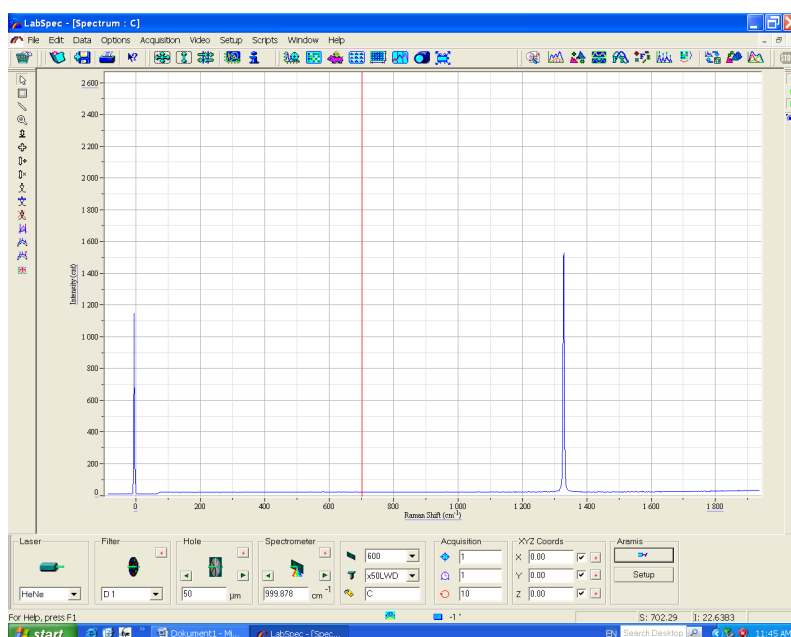
Use the settings in the menu on the left side of the screen (Picture 10) to obtain an optimal signal.

The resulting images should look like those in Pictures 10 (for silicon) and 11 (for diamond).



Picture 10. Raman spectrum for Si. Indicated: save spectrum (File), Acquisition and picture settings menu (at the top) and microscope aperture icon (at the bottom).

15. Save the spectrum by clicking “File” in the main menu and selecting “Save as”.
Save the spectrum in two files: one in *.ngs format; the second as *.txt (txt format is suitable for use in Microcal Origin and MS Excel).
16. Turn off the spectrometer. To do this, close the microscope aperture with the “Hole” dialogue in the measurements menu. In the main menu under “Acquisition”, enable “Heat detector”. Wait until the temperature detector rises above 0°C.
17. Turn off the computer. Turn off the spectrometer (using the switch on the power strip).
18. Develop the results:
 - a) present the Raman spectra for Si and C in a single graph;
 - b) discuss the energy of the major spectral lines obtained using the optical phonon model in crystals.



Picture 11. Raman spectrum of diamond.

III. Apparatus.

1. LabRam Aramis Raman spectrometer from Horiba Yobin Yvon.
2. Synapse CCD camera from Horiba Yobin Yvon.
3. Control joystick for the microscope’s adjustable sample table.
4. Two microscope illuminators for transparent and opaque samples.
5. Transformer.
6. Sample table power supply.
7. CCD camera power supply.
8. He-Ne and YAG:Nd lasers’ power supplies.
9. Computer.

IV. Literature.

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