Optical Infrared High-Pressure and Low Temperature Setup

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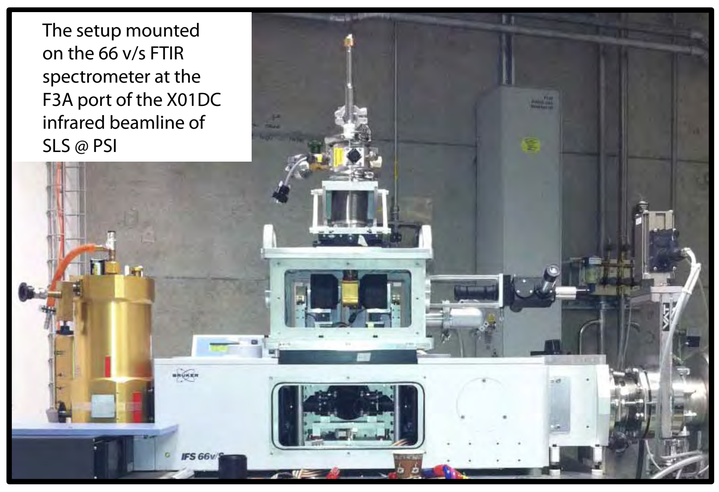
Temperature-dependent optical infrared spectroscopy allows to probe low energy vibrational, electronic and magnetic excitations present in a large variety of systems. Applying pressure, provided hydrostatic conditions are obtained, modifies the inter-atomic distances (if not the crystal structure of the material). Therefore, pressure can be thought as a «dial knob» of the electronic band structure of materials. Thus, interactions between charge carriers can be probed, without the often-unknown effects present when chemical doping is used instead.



The setup mounted on the 66 v/s FTIR spectrometer at the F3A port of the X01DC infrared beamline of SLS @ PSI

Main components

The setup is essentially an infrared microscope working entirely in vacuum. It's based on three essentials parts:

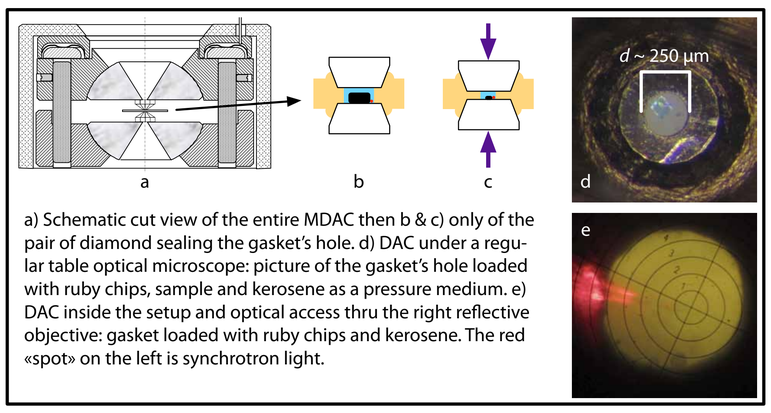
- A BETSA Diamond anvil pressure cell
- A Janis ST-100 helium-flow cryostat
- A Bruker 66V Fourier-Transform Infrared Spectrometer coupled to the IR synchrotron source.

Samples

- Liquids
- (Very) small crystal samples or powders + a transmitting medium are loaded into a hole drilled in a CuBe gasket along with ruby chip(s) as a pressure gauge.

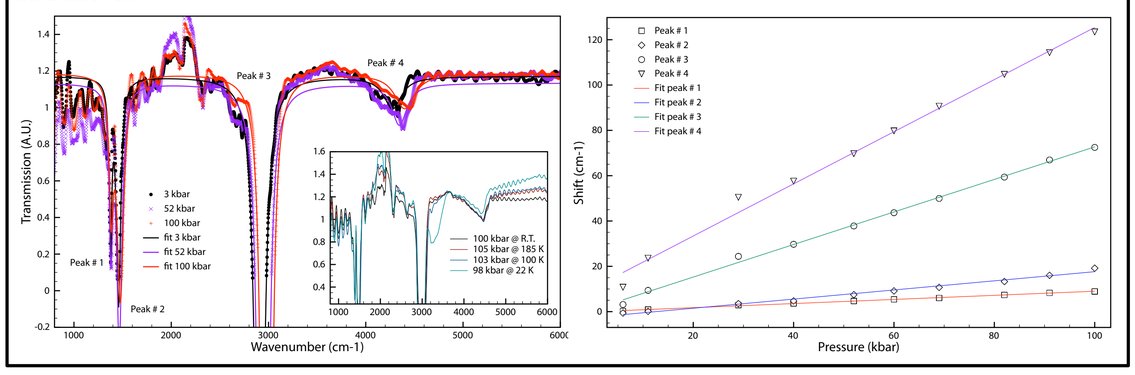
Experimental constrains

1. Measurement in transmission mode
2. Targeted pressure range defines sample's size
3. Limited observable range in the far infrared (d>5 λ)
4. Reduced optical throughput is compensated by the use of a synchrotron light source
5. When used, possible low to mid infrared absorption the pressure-transmitting medium
6. Sample cannot be thicker than the gasket (~50µm)
7. Strong absorption in the 2000 - 2500 cm^{-1} range due to the two diamonds type IIa of the DAC



a) Schematic cut view of the entire MDAC then b & c) only of the pair of diamond sealing the gasket's hole. d) DAC under a regular table optical microscope: picture of the gasket's hole loaded with ruby chips, sample and kerosene as a pressure medium. e) DAC inside the setup and optical access thru the right reflective objective: gasket loaded with ruby chips and kerosene. The red «spot» on the left is synchrotron light.

Example: spectra of kerosene under high pressure and at low temperature. A shift of the fitted peak positions (R.T. only) towards higher frequencies is observed.



Setup and optical path

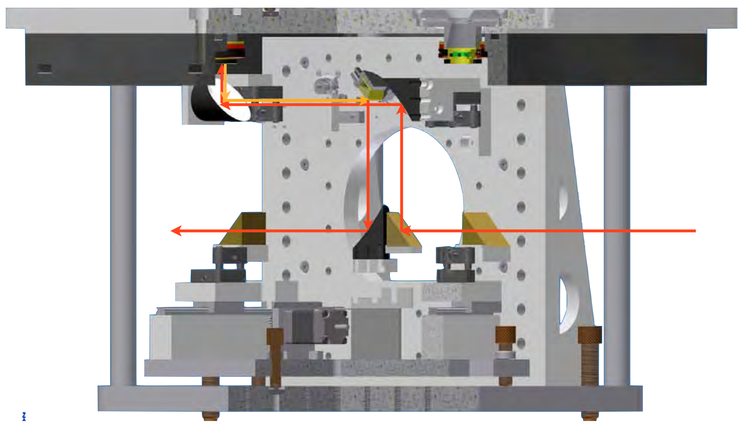
Within the spectrometer's sample chamber a plane mirror deflects the infrared beam through KBr sealing windows to an upper chamber, were another plane mirror steers light into a 15X magnification reflective Cassegrain objective. The sample mounted into the DAC which is itself attached to the cold finger of the Janis cryostat, can be adjusted into position by an XYZ stage. Transmitted light is collected by a another Cassegrain objective and send to the detector along a symmetrical optical arrangement. Sample temperature is measured at the end of the coldfinger as well as within the pressure cell. Temperature regulation is achieved by adjusting the helium flow together with the electrical power dissipated in a cartridge heater attached to the cold finger.

The pressure in the diamond anvil cell is tuned in situ by the use a helium-inflated membrane connected to a capillary network. The optical path can be changed by a motorized mirror/beamsplitter allowing optical access to the sample through quarzt windows. The pressure is determined by the shift in wavelenght δλ of the R_1 peak in the ruby's fluorescence spectrum.

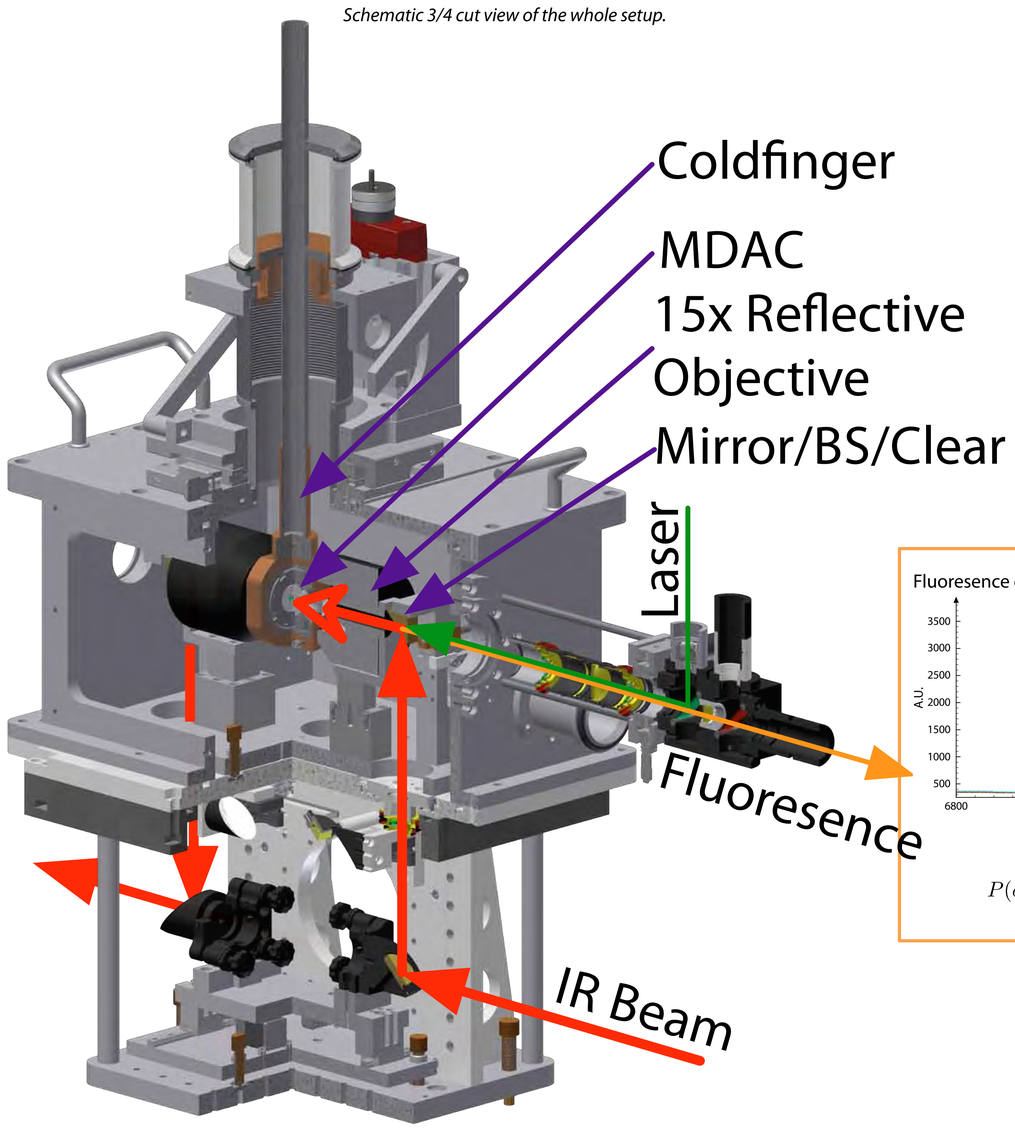
- Spectral range: from far infrared (depending on sample's size) to visible & UV (with differents detectors)
- Temperature: from room temperature to **22 K**
- Pressure: from a few kbar to **200 kbar**

Outlook: performing reflectivity measurements

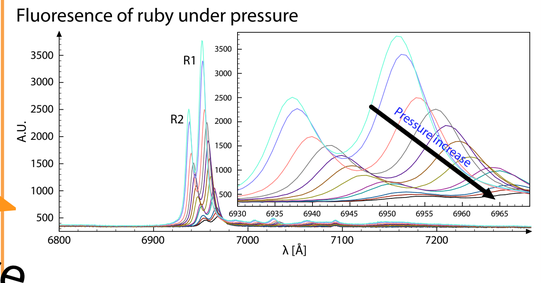
Schematic 3/4 cut view of the IFS 66 v/s sample chamber with a switching optical path for transmission and reflectivity measurements



Schematic 3/4 cut view of the whole setup.



Fluoresence of ruby under pressure



$$P(\delta\lambda, R_1, B) = \frac{1904}{B}\left[\left(1+\frac{\delta\lambda}{R_1}\right)^{B} - 1\right]$$

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