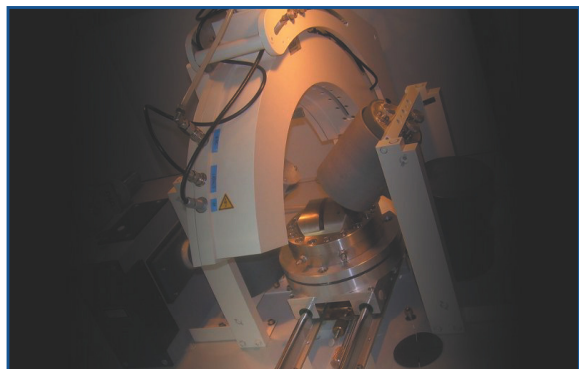


Combined Technique Instrument

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Context : The study of the dynamic behavior of materials is complex, since it often requires several instrumental techniques to understand the physical and chemical processes. With complex and not understood systems, the concept of reproducibility is important. The number of parameters, controlled or not, is often too important. This means that several measurement instruments, including X-ray diffraction, will be set up around a single cell sample, to be followed in real time the evolution of structural modifications. The effects of changing various parameters (pressure, temperature, gas flow ...) will then be directly recorded by all instruments.

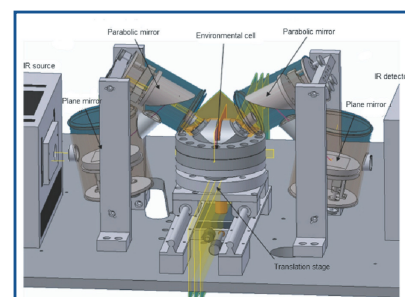
Operando XRD-DRIFT : Catalyst characterization by Fischer-Tropsch process for clean fuel production

$$n\text{Co} + 2(n+1)\text{H}_2 \rightarrow \text{C}_n\text{H}(2n+2) + n\text{H}_2\text{O}$$


INEL has designed a transmission mode diffractometer with a curved position sensitive detector associated to a DRIFT spectrometer. The system includes an environmental cell that allows X-ray and IR analysis at high pressure and high temperature.

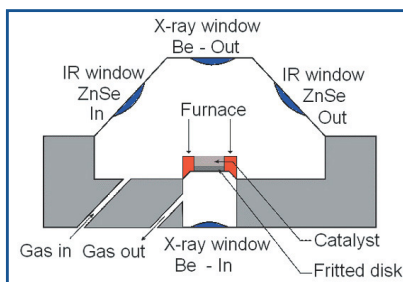
XRD specifications :

- Transmission mode with a Mo tube ($\lambda = 0,709 \text{ \AA}$ - $E=18\text{keV}$)
- Inel CPS120 curved detector, (Acquisition in real time over $120^\circ 2\theta$, radius : 250 mm)
- Focusing optic
- Debye-Scherrer geometry



DRIFT specifications

- IRcube OEM FT-IR spectrometer
- Set of parabolic and plane mirrors



Cell specifications

- Gaseous reactants flow up and down the reaction cell through a fix sample
- Heavy products collected in a condenser cooled at 15°C
- Operating conditions (T/P) : up to 600°C / up to 18 bars
- Specific windows for each experimental equipment

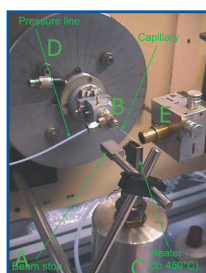
→ Connected to a gas chromatograph for on-line analysis

Thanks to V. Moizan-Baslé, L. Braconnier, I. Cléménçon, C. Legens, B. Rebours, L. Lemaitre, IFP-Lyon, BP 3, 69360 Solaize Cedex, France

Innovative system for the in situ investigation of hydrothermal reactions by X-ray diffraction

Specific instrumental device :

- A purpose-built capillary reaction vessel which allows close emulation of the conditions present in mineral processing plants
- Transmission mode with the Mo K radiation, to ensure that the X-ray beam penetrates through the capillary
- An Inel CPS120 position sensitive detector, to enable simultaneous collection of a wide range ($120^\circ 2\theta$) of diffraction data



- A : beam stop
- B : sample holder (including the Swagelok settings to allow the application of gas pressure) and capillary reaction vessel
- C : nozzle of the heater
- D : pressure line
- E : the incident-beam slit holder

A close-up view of the sample configuration

Capillary mounted on a head goniometer

Heat zone : heating hot jet - T up to 750°C

Pressure applied with a sweep gas (CO_2 , N_2 , H_2 , O_2 , Ar, etc.) - P up to 50 bars

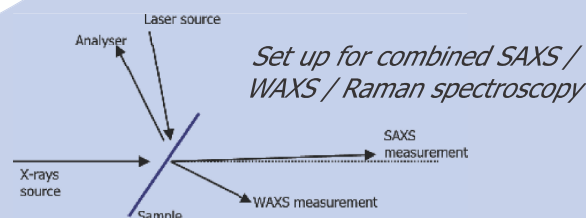
Thanks to Ian C. Madsen, CSIRO Minerals, Box 312, Clayton South, Victoria, 3169, Australia

SAXS WAXS experiments combined with a Raman spectrometer for thermal studies

Proteins are essential components of all living organisms. Involved in many biological processes, they may be therapeutic targets and as potential drugs. One way to characterize them would be to develop innovative systems using Raman spectroscopy (or FTIR) coupled with X-ray scattering to study in situ the structure of proteins in solution (emulsion) or solid state (lyophilized powder) while having the ability to optimize the operating conditions (P, T, pH, ...).

Specific instrumental device :

- Transmission Mode with a Cu tube ($\lambda = 1,54 \text{ \AA}$ - $E=8\text{keV}$)
- An Inel CPS120 curved detector for WAXS measurement + a 2D detector or an Inel linear detector for SAXS measurement
- In situ reaction with a heating hot jet or/and heating cold jet



Set up for combined SAXS / WAXS / Raman spectroscopy

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