



NRC-CNRC

*Institute for
Chemical Process
and Environmental
Technology*

Science with a novel gas pressure (CO₂) apparatus

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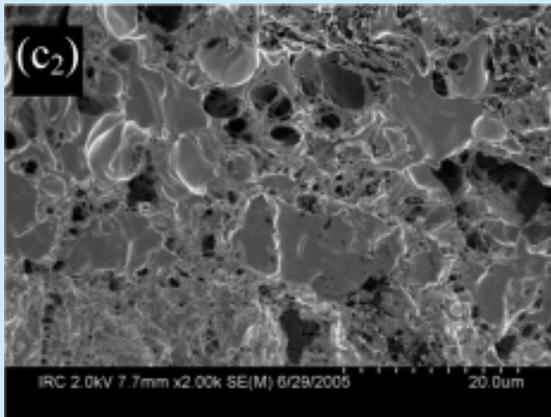
National Research
Council Canada

Conseil national
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Canada

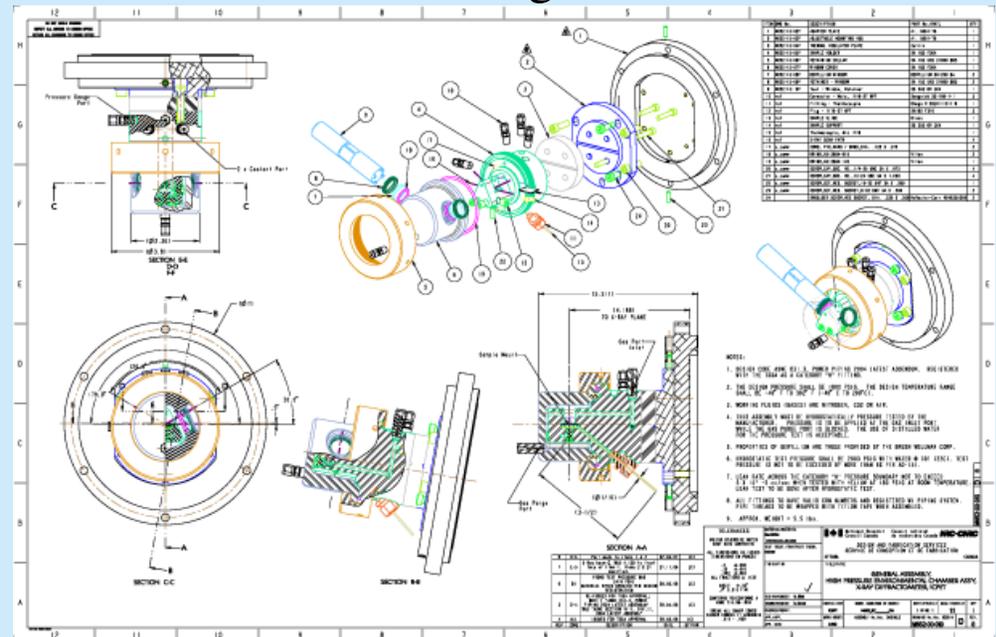
What was our interest?

- Interested in the use of sub and supercritical CO₂ for polymer processing / microstructure control (crystallization, foaming, etc)
- Compact lab XRD in-situ gas cell designed and built for CO₂/inert gases up to 125 bar (-40 to 200°C)



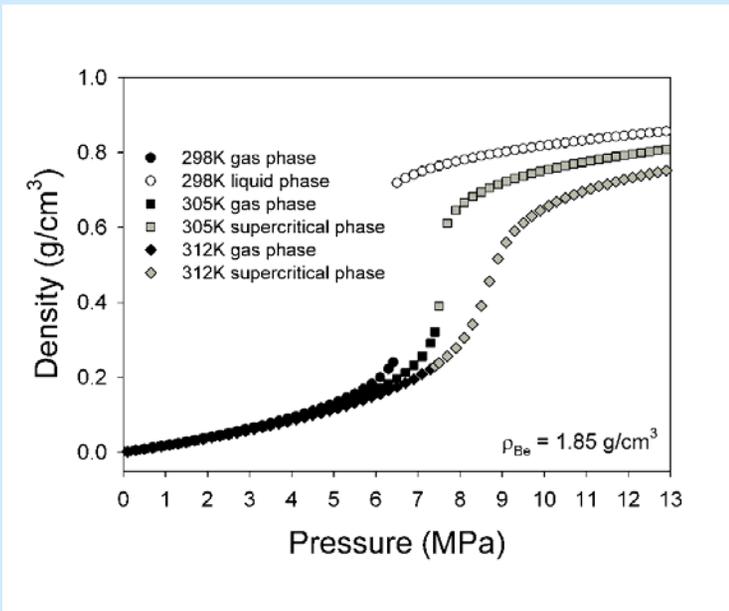
Foamed PLLA produced using 5.5 MPa CO₂ at 90°C for 5 mins

CAD drawing of the cell

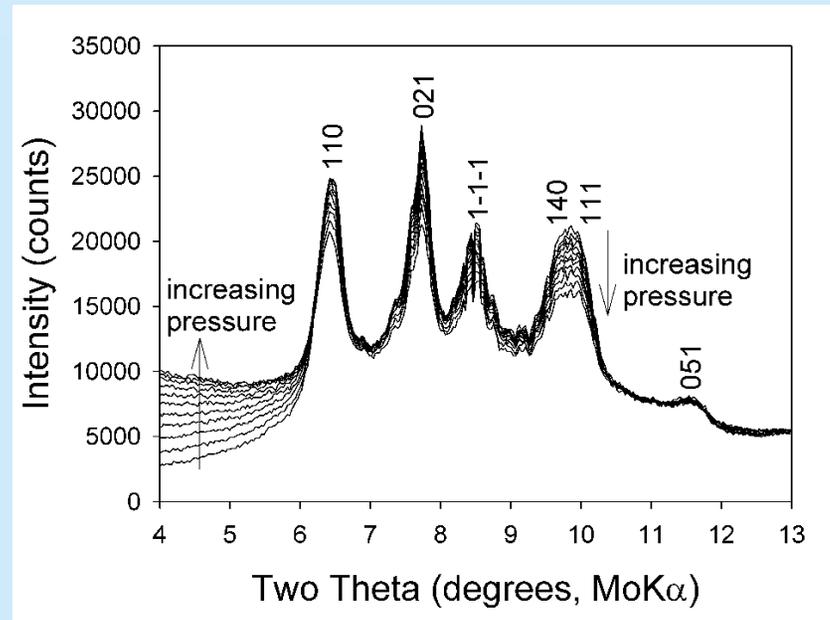


More pressure stage...

- Design subjected to finite element analysis, certification and testing
- Completely legal!
- Pressure/temperature/density characteristics of CO₂ particularly nasty
- Close to liquid boundary small changes in stage (or other equipment) temp has large effect on pressure/density



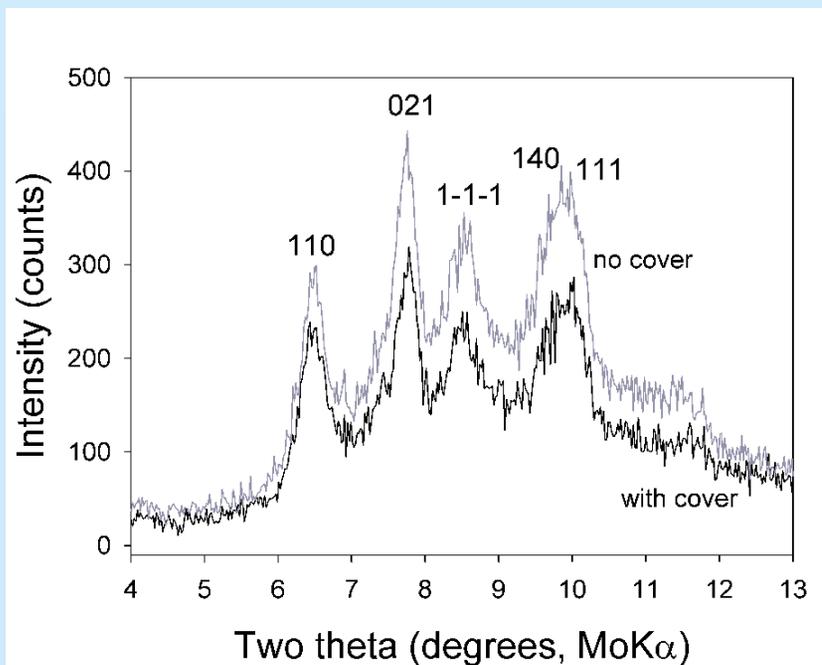
Vapour/liquid/supercritical boundaries



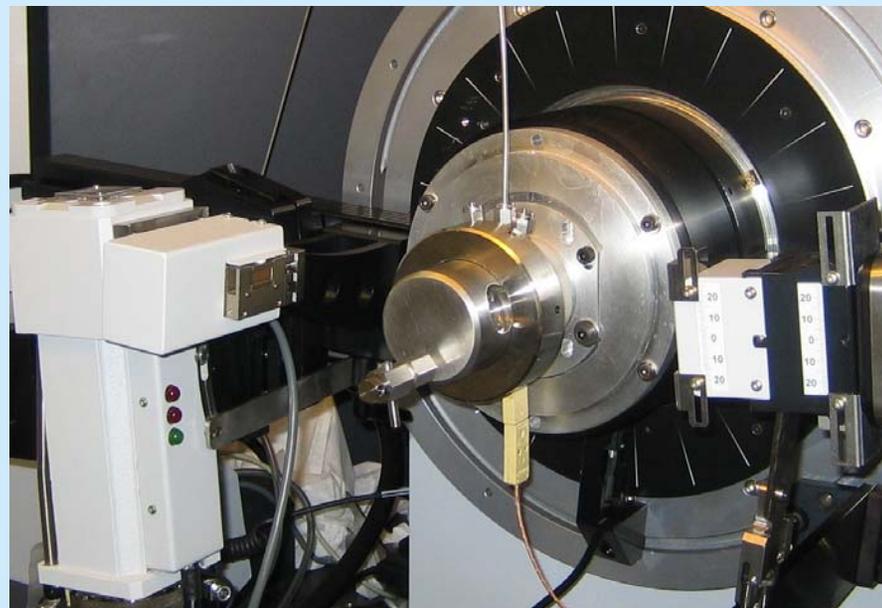
Polypropylene snapshots 0-1000psi

How far can I go in the lab?

- Coupled with Mo tube and Vantec PSD detector in fixed scan mode can do kinetic studies with time resolutions of the order of seconds
- Haven't had chance to hook up recirculating fluid heating/cooling yet



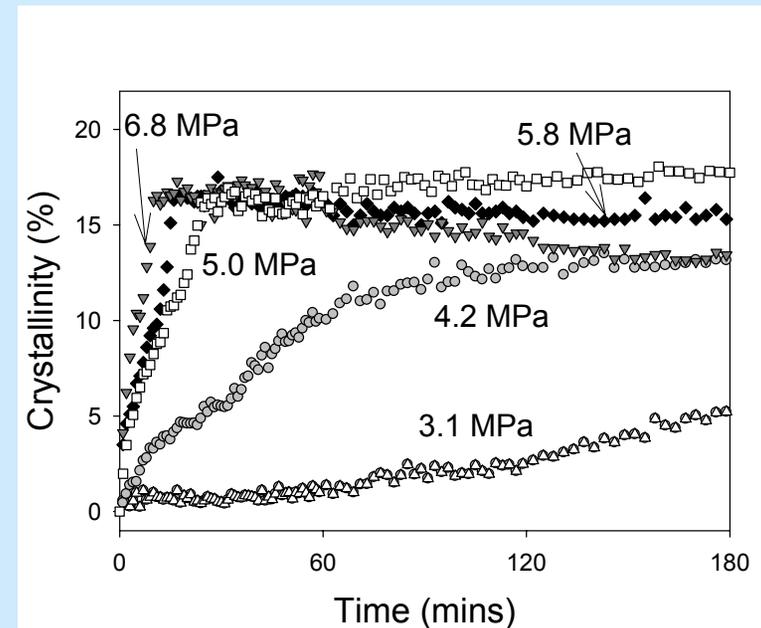
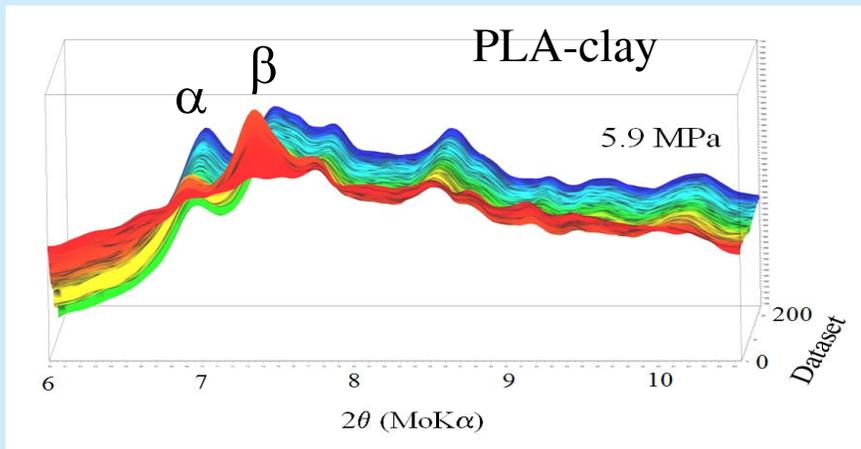
1s snapshots of polypropylene at 1atm



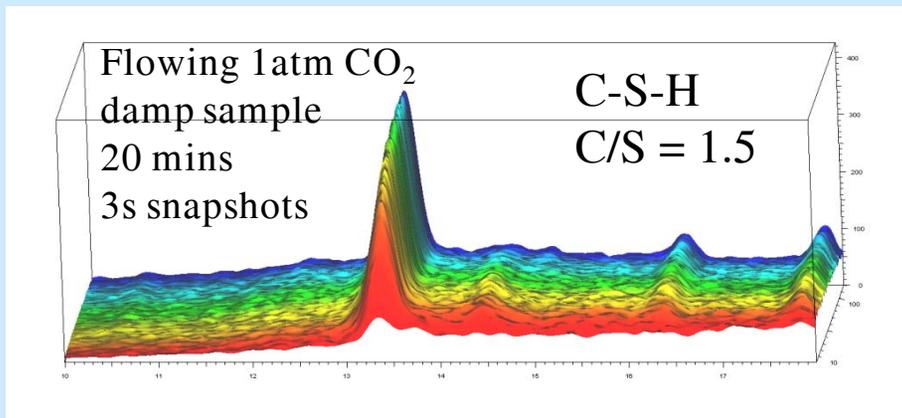
Cell mounted on Bruker D8

What's been done in the lab so far...

- Crystallization of polylactic acid (PLA) and PLA-clay composites
- Carbonation of calcium silicate hydrate (cement binding phase)



Crystallization over the first hour for PLA-clay nanocomposite at different CO₂ pressures @29°C

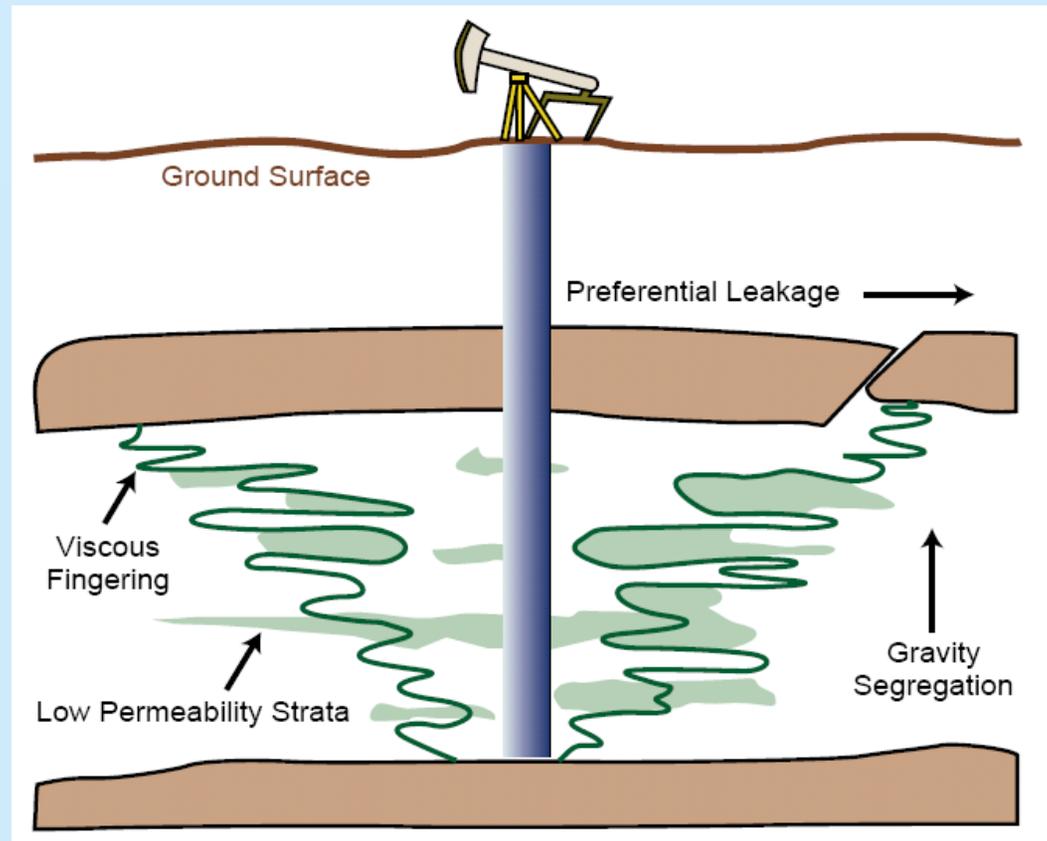


The future?

- If time resolution of seconds possible with a lab Mo source what could NSLS-II achieve?
- Current stage possible basis for a third generation custom gas cell for NSLS-II
- Can optimize design for high energy
 - No need for Be windows
 - Higher pressures easily achievable
 - Less attenuation at higher energies
 - Energy/brightness → Better time resolution for kinetic studies
 - Add flammable gas certification

Potential research areas – CO₂

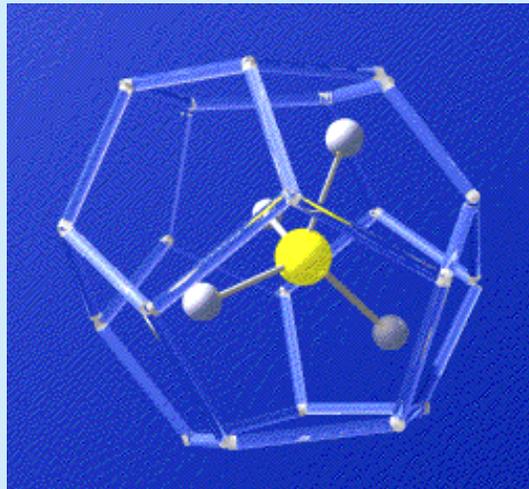
- Materials processing in supercritical CO₂
 - Polymers, extraction, etc
- CO₂ sequestration mineralogy studies
 - Rock/ CO₂ interactions
- Flammable gases
 - CO₂/CH₄ mix
- No Be corrosion
 - CO₂/H₂O mix



Other systems

- Hydrogen storage (low/medium pressure, 100-400°C)
- Gas hydrates (low pressures, sub-ambient temps)

Clathrate structure



- Organic magnets (would need kilobar pressure?)
- Anything anyone else can think of...
- Suggestions?

- All sorts of interesting applied and fundamental science possible with quite moderate gas pressures
- CO₂ is an awkward system that can require very careful control of pressure/temperature conditions to remain stable
 - Increase in temp of just few degrees can increase liquid boundary by 10s of PSI
 - Have to think about source/reservoir conditions as well as stage itself
- Some cool stuff has been done using a lab setup in a matter of weeks
 - Imagine what you could do using PING? 😊