High-throughput Methods for Material Science Research in Energy Applications

NCMC-14: DOE-EERE/NIST Joint Workshop on Combinatorial Materials Science for Applications in Energy

November 6, 2008

Symyx

Outline

- Overview
- Polymeric Materials Examples
- Inorganic Materials Examples

Overview

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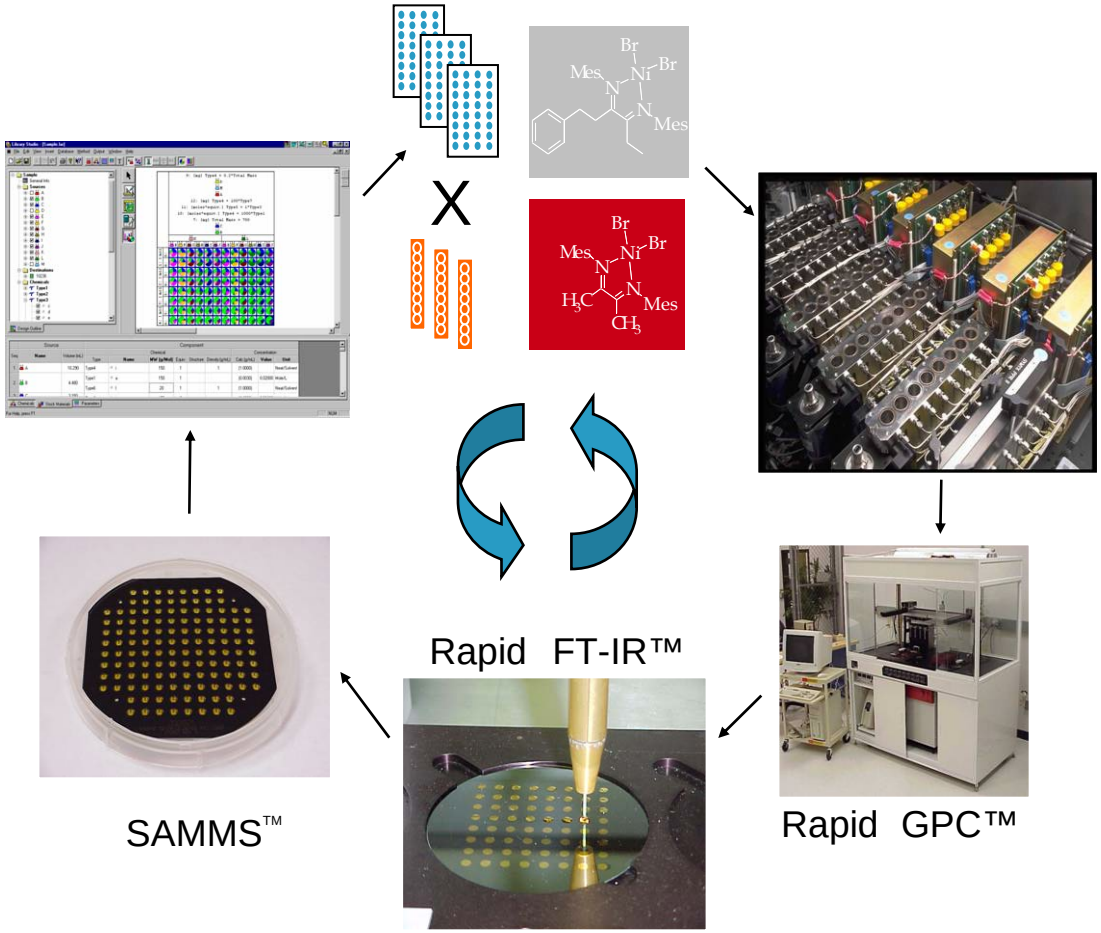
The Product Development Challenge

Most customers don't buy *molecules*, they buy *combinations of properties*

What does "classic" high throughput research *…have to do with* **this***?*



Symyx polymer catalysis discovery workflow

Charpy impact tester for failure measurements of solid polymer specimens

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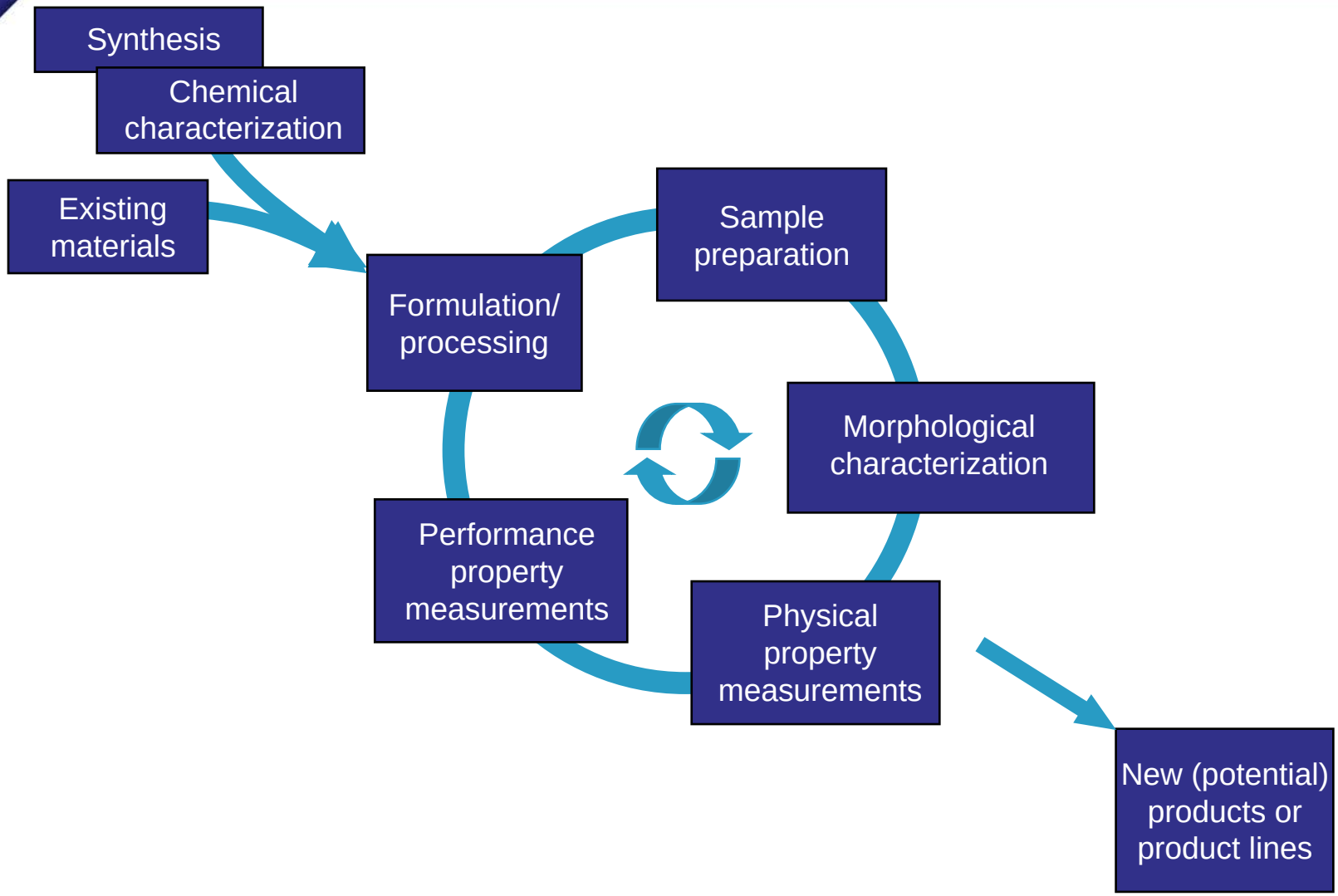
Accelerating the Complete Workflow



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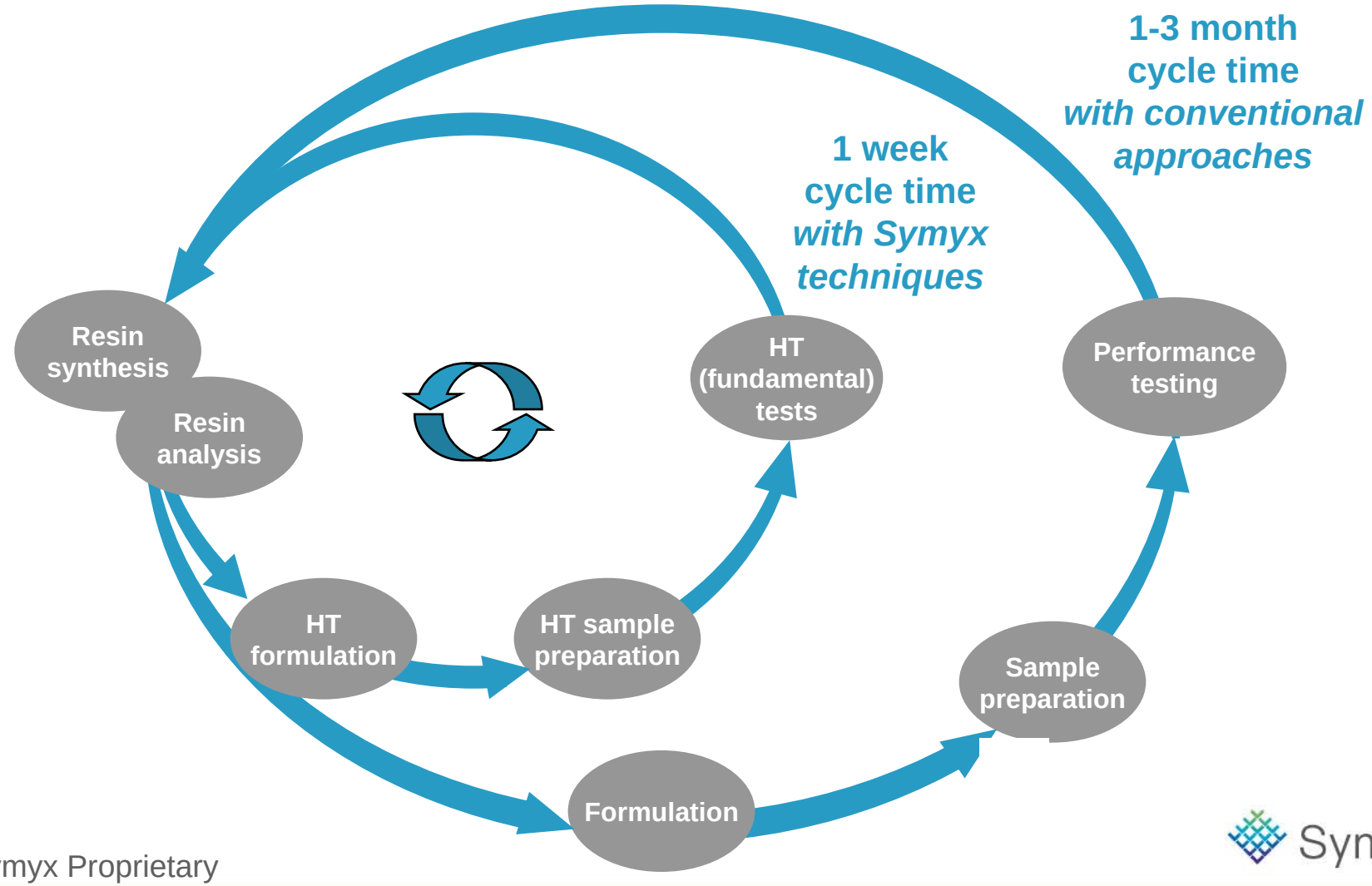
Addressing the Performance Testing Bottleneck

Fast (inner) research cycle screens for performance on slow (outer) research cycle



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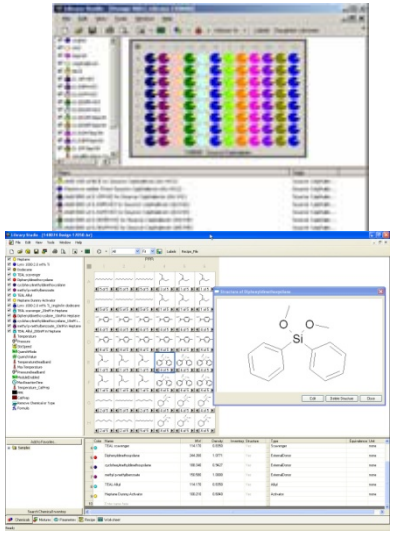
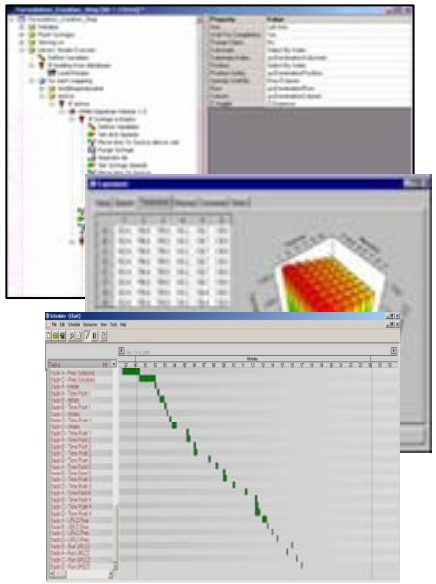
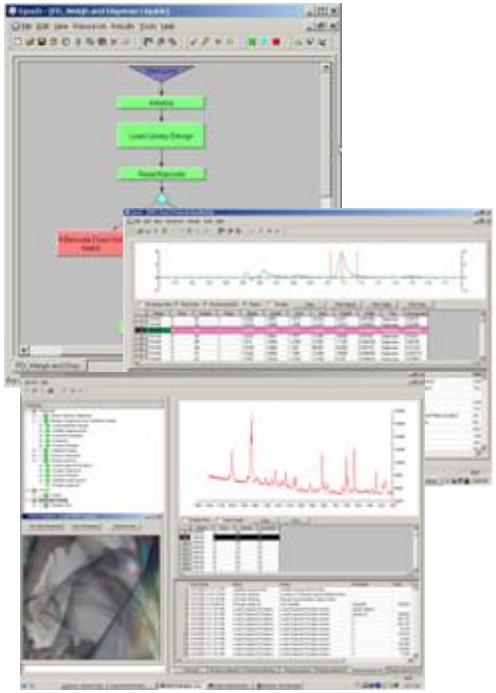
Software is the Backbone of any Workflow

Design	Execute	Analyze	Report

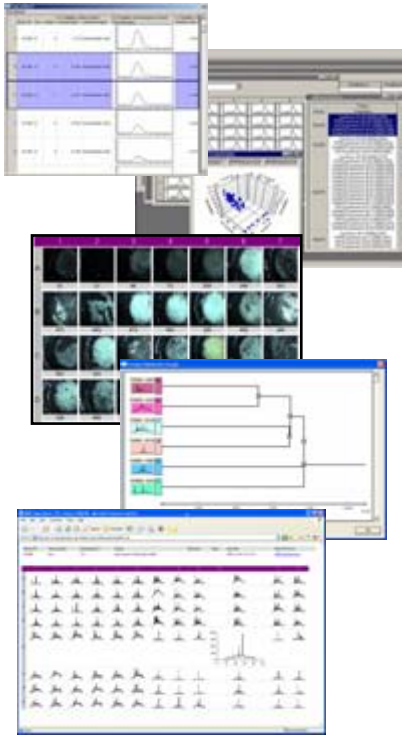


- Experiment Design





- Instrument & Experiment Control



- Lab Analysis and Reporting

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Polymer Materials Examples

Polymer Synthesis, Blending and Property Measurement

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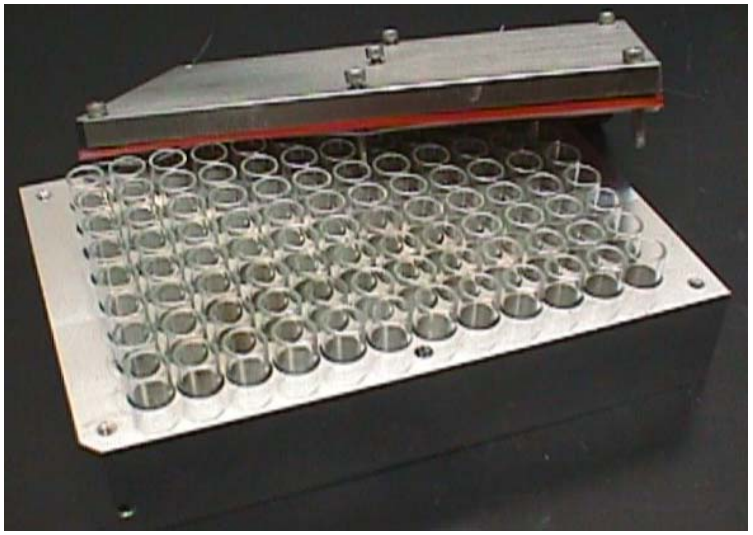
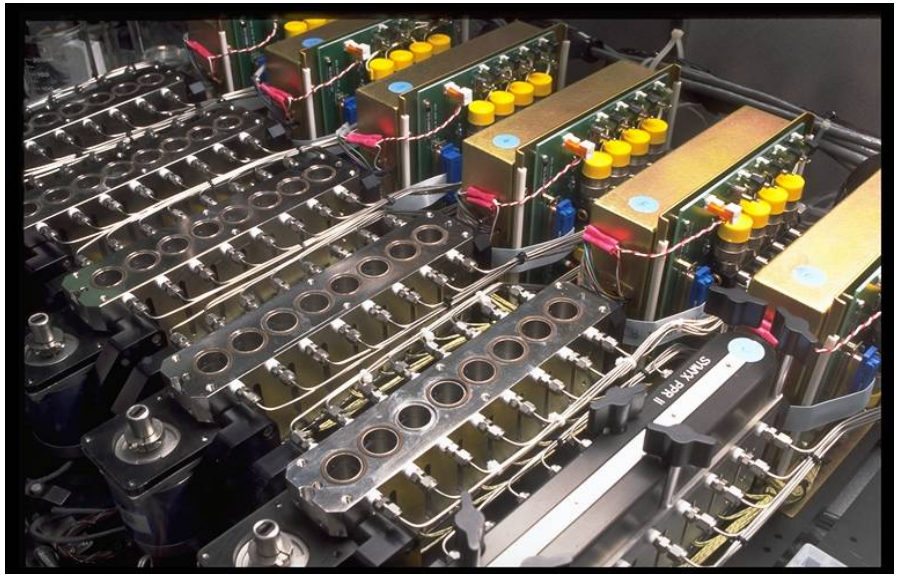
Typical Polymerization Reactors

Batch parallel reactor- Primary Screen

- 96-well glass-lined aluminum microtiter plate with PTFE-faced silicone seals (800 μl scale)
- Compatible with organic and aqueous solvents at elevated temperatures, pressures
- Agitation (steel balls, stir bars)





Batch parallel reactor- Secondary Screen

- 48-well glass-lined reactors with gas and liquid delivery through pressurized ports (5 ml scale)
- *In situ* characterization of reacting mixtures
- Mechanical agitation (impellers)

Semicontinuous Synthesis

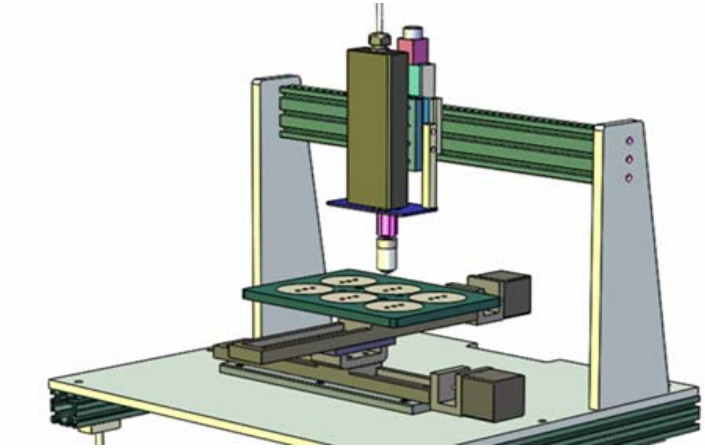
- Semicontinuous processes common in industry for control of
  - Relative reactant concentrations
  - Monomer sequence
  - Monomer starvation (for kinetics, safety)
- Equipment features
  - Three 8-cell strips (24 reactions, 4 to 10 ml)
  - Up to 12 feed lines with independent inlet heights
  - Mechanical stirring
  - High pressure capabilities (1200psig)

Typical reaction volume of 3 to 6 ml yields 0.25 to 2.0 g of polymer in each of 24 cells

Symyx Proprietary

Chemical Characterization

Spectroscopy

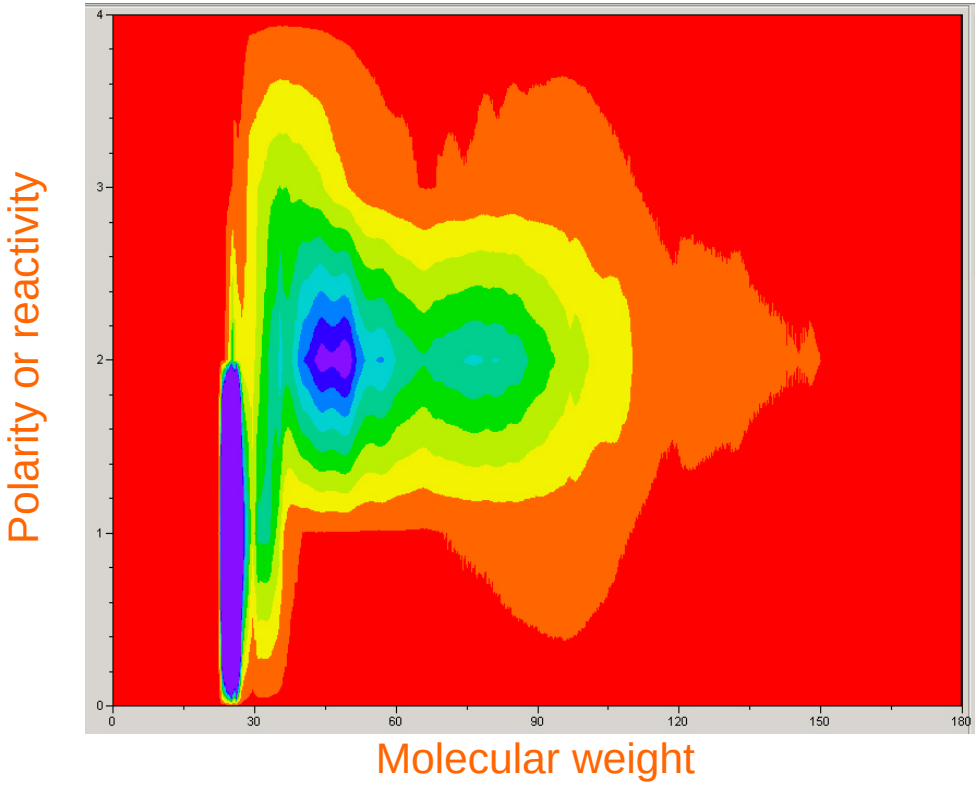


- High throughput versions of FTIR, Raman, visible, and UV spectroscopy

Chromatography

- Separations by most (common) mechanisms in 1-10 minutes/sample
- Multidimensional separations

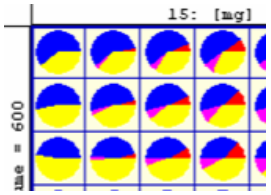
Multidimensional chemical characterization



- Output aliquots from first analysis technique passed immediately to second analytical technique
- Provides “fingerprint” of complex mixtures
- Data can be generated at multiple wavelengths for more detailed analysis of chemical composition

High Throughput Melt Processing

Symyx software
(Library Studio ®)



Design arrays of blends to be made

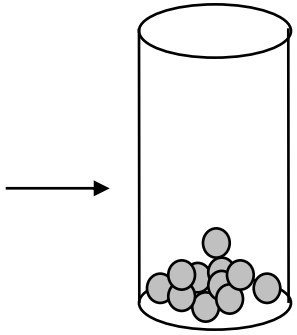
Liquid handling robot

Powder handling robot

Weigh out blend ingredients into vials

Automated microcompounder

Automatically deliver, mix, pelletize, and collect polymer blends



Output: array of pelletized polymer blends

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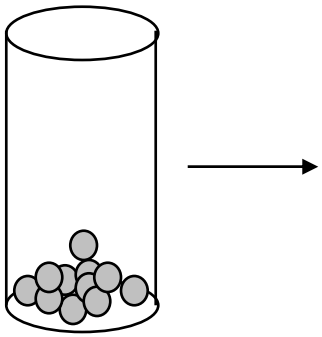
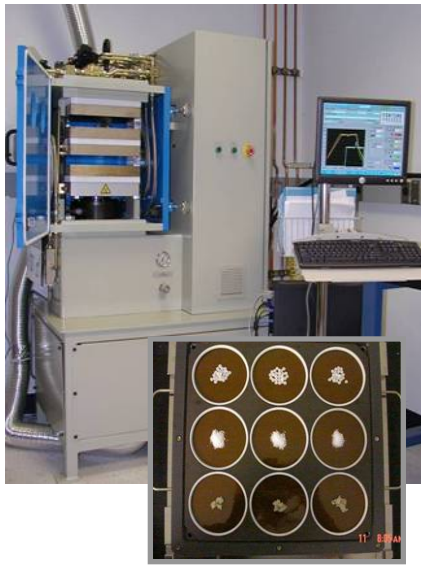
High Throughput Melt Processing

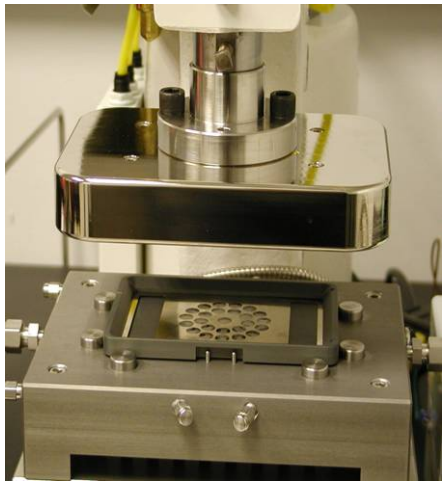


Multilayer vacuum press (MVP)



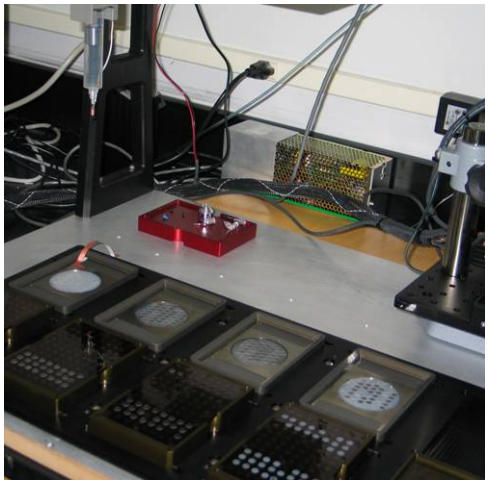
Melt and press up to 18 samples into uniform discs under vacuum

Die cutter



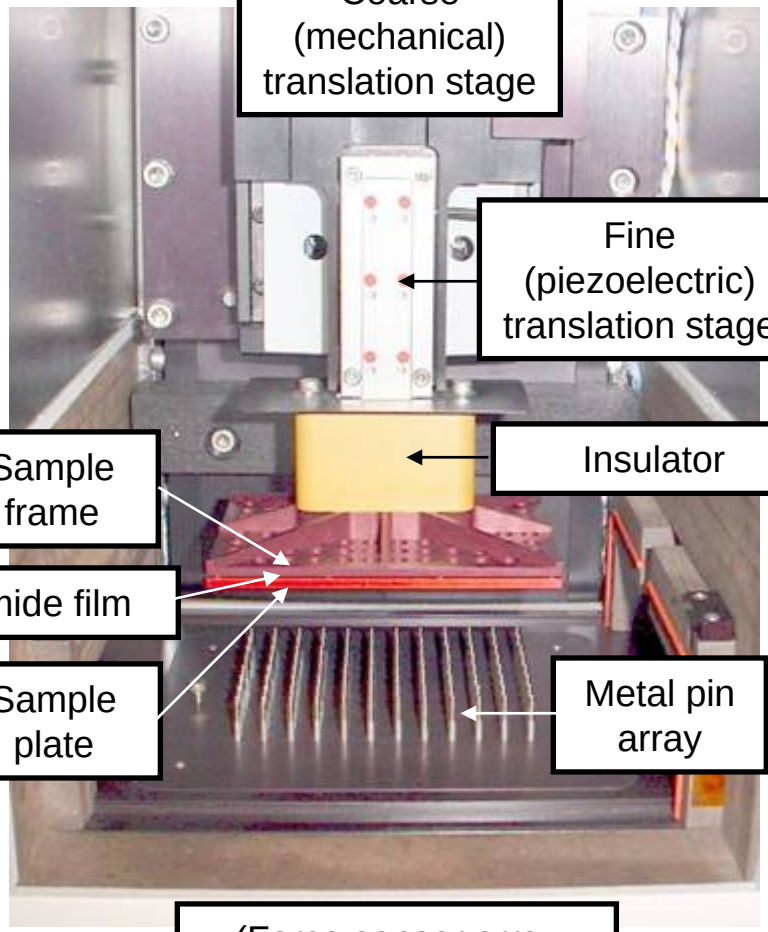
Die-cut polymer sheets into appropriate shapes dictated by the tests to be run

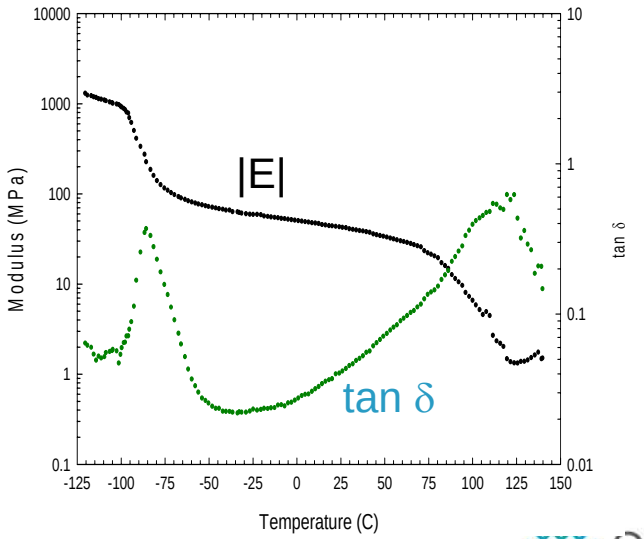
"Pick and place" robot



Automatically transfer each sample to a variety of substrates (e.g. DSC pan, DMTA plate)

Parallel Dynamic Mechanical Characterization





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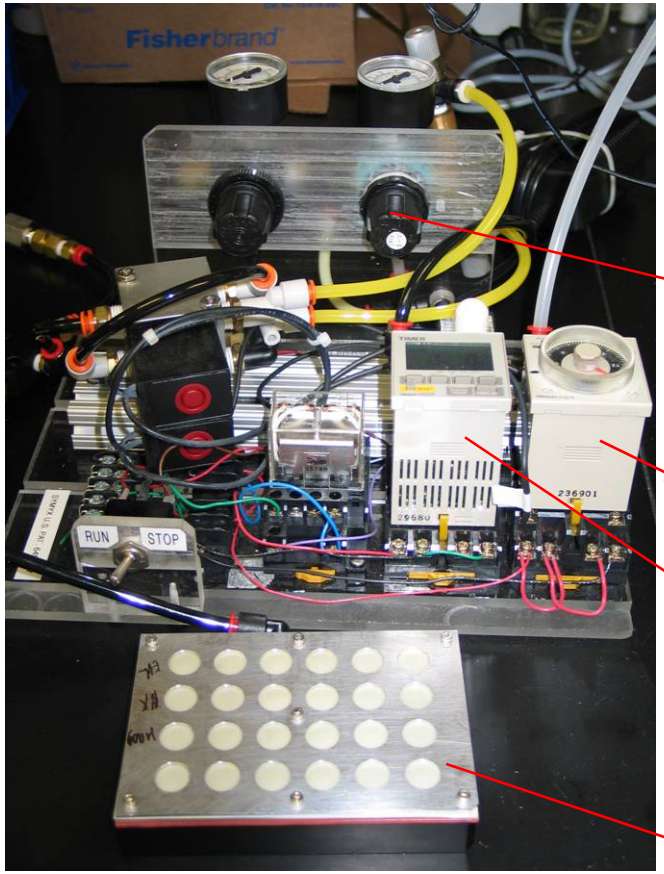
Flexural Durability (small strain)

Flexural durability tester



Pressure controller

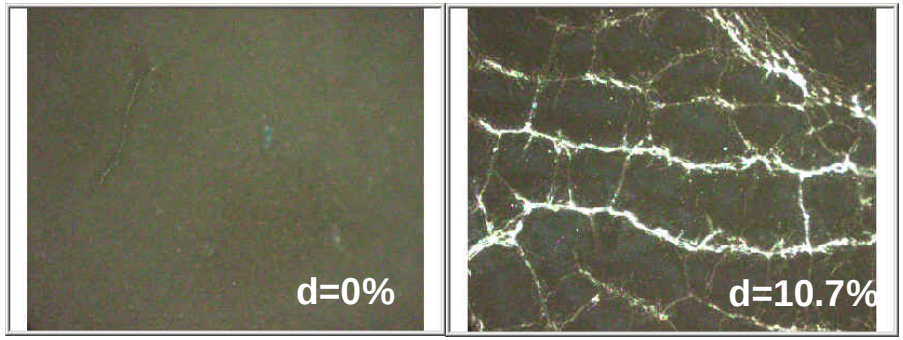
Frequency controller

Timer

Sample assembly

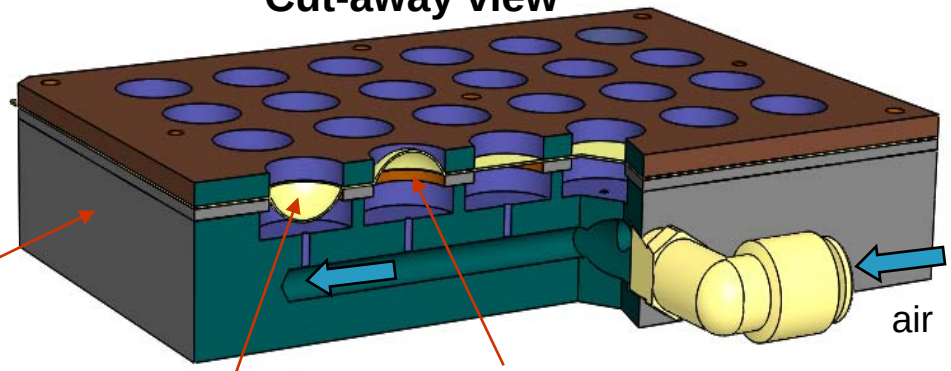
Microscope images

Non-cracking *Cracking*



d: Crack ratio

Cut-away view



air

With vacuum

With pressure

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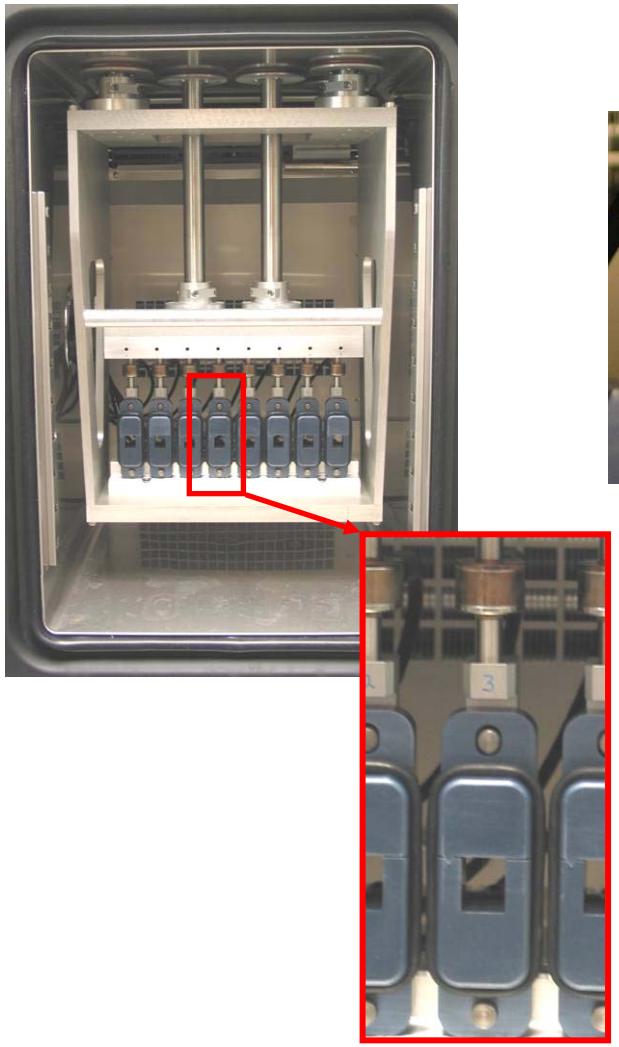
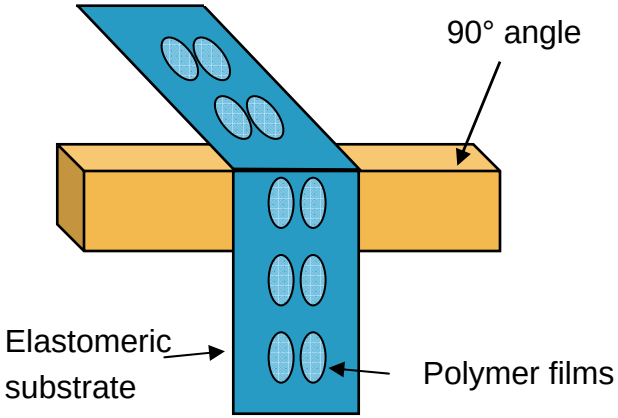
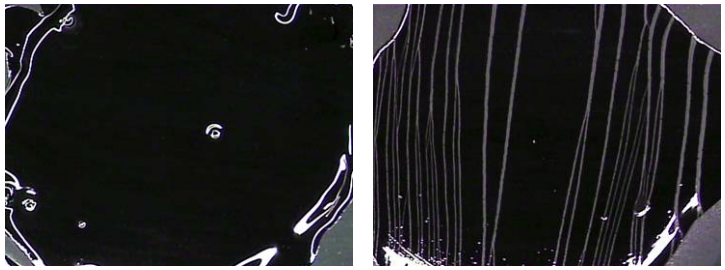
Flexural Durability (large strain)

Parallel elongation-to-break

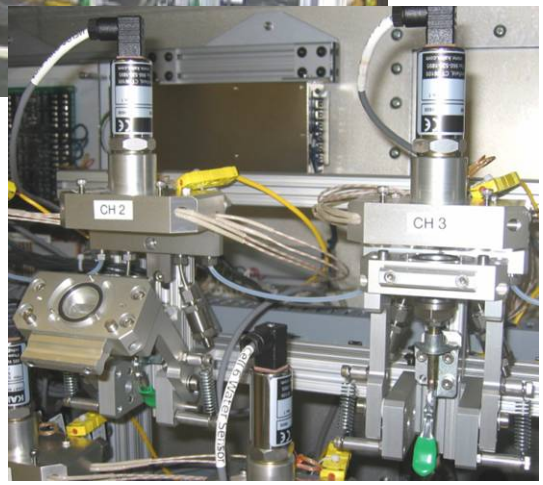


Parallel sharp bend





Permeability



- 24 samples individually clamped between seals and exposed to common feed
- Sensor monitors rise in permeate concentration on opposite side of membrane

Inorganic Materials Example

Inorganic Synthesis and Property Measurement

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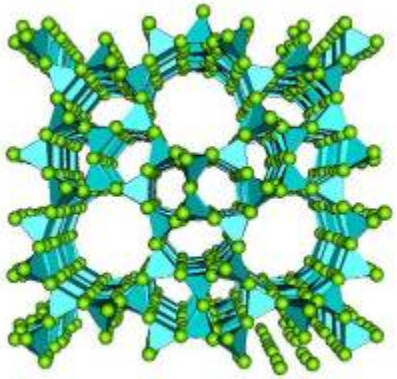
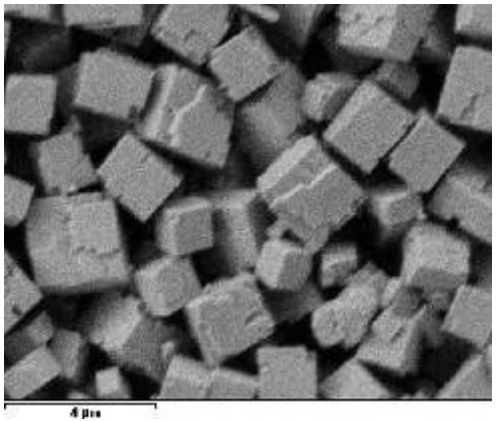
Hydrothermal Synthesis

- Zeolites, mesopores, MOFs
  - Parallel reactors from 100 uL to 25 mL volume
  - Gel prep station based on Core Module robotic platform
  - Particle recovery system
  - SEM, XRD, TGA, surface area, pore volume, PSD

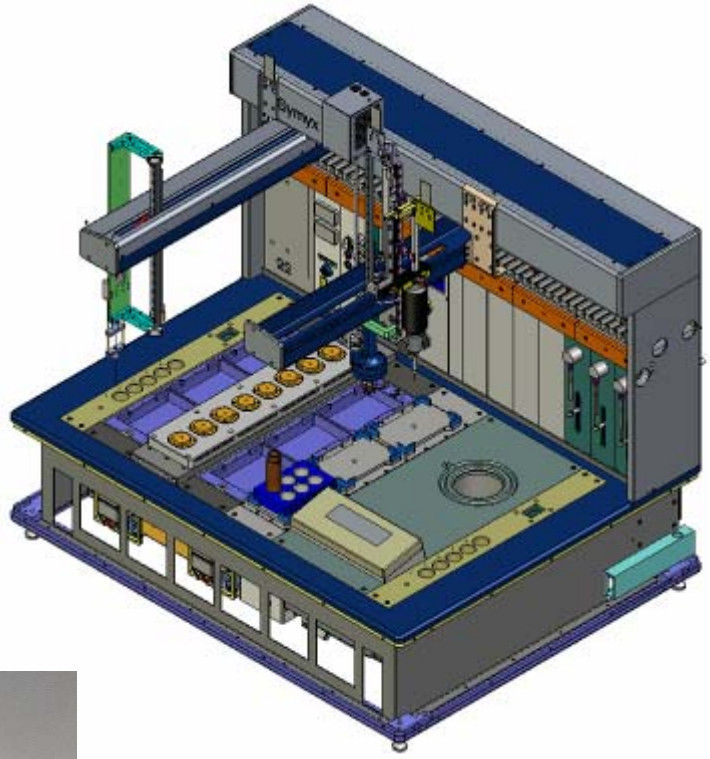




Parallel Pressure Reactor

Nanoparticle Synthesis

- System designed to flexibly automate synthesis of semiconductor nanocrystal "quantum dots"
  - Viscous reagents are weighed, mixed and preheated, then injected into 350 ºC reactor with stirring under vacuum or purge gas. Aliquots can be extracted at defined intervals. Reaction can be rapidly quenched.

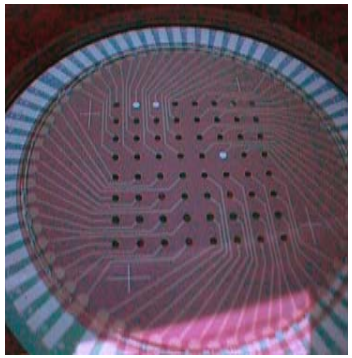


Core module with LTMR, viscous liquid dispense, heated mixers, balance, and reagent injection or sampling.

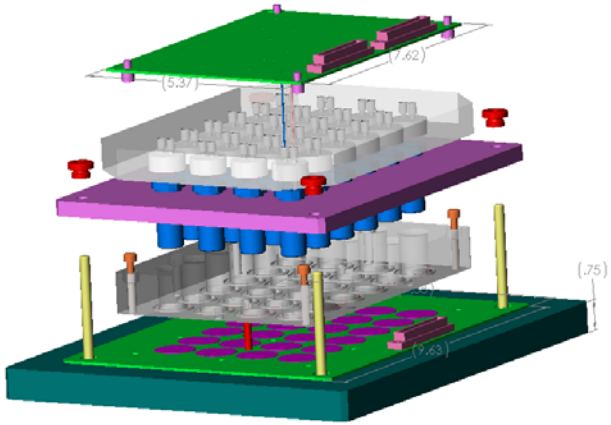
CdSe quantum dots

8-channel Low Thermal Mass Reactor (LTMR)

Fuel Cell and Battery E-chem Techniques

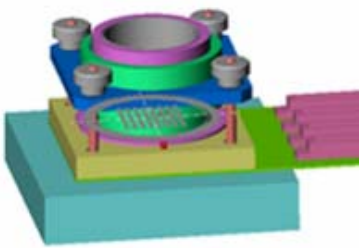


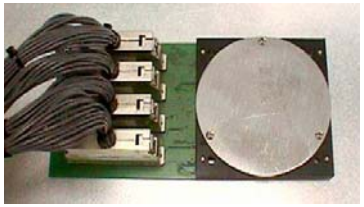
Addressable electrode array and electrochemical cell with multi-channel potentiostat

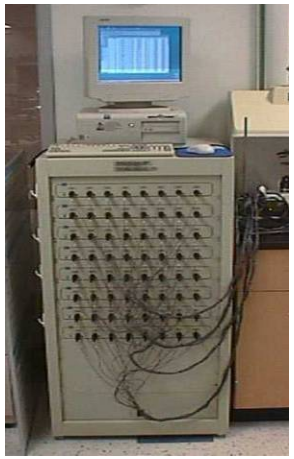


24-channel independent electrochemical cells

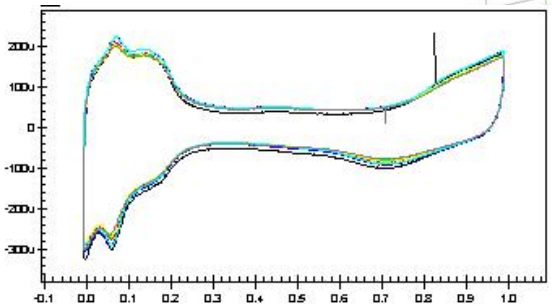
8- and 16-Parallel Rotating Disk Electrode Systems

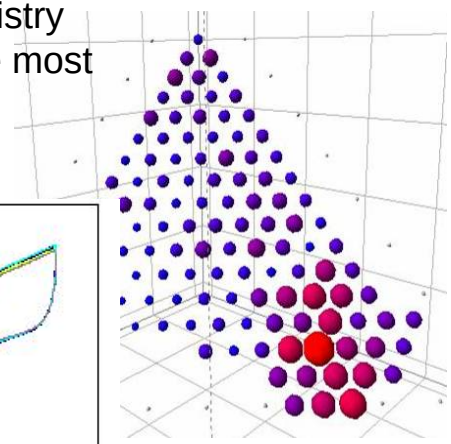






High throughput electrochemistry from primary screening to the most sophisticated measurements





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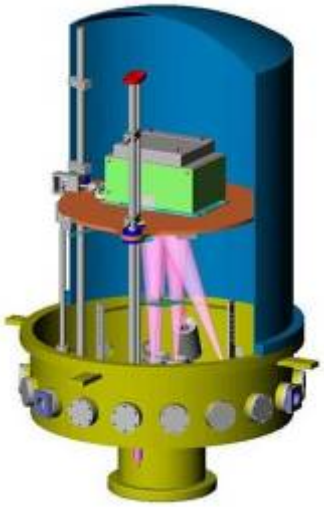
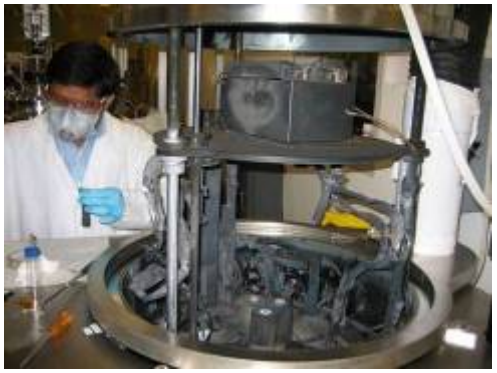
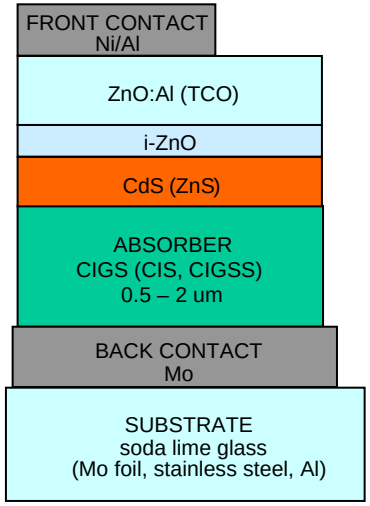
Solar Cell Process and Characterization

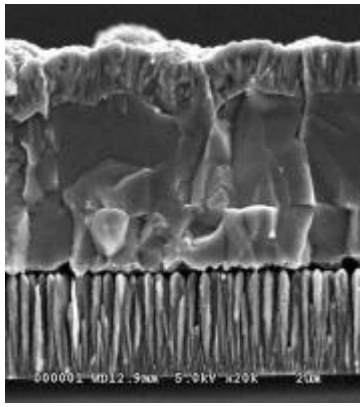
Chemistry to process to device. Multiple, highly varied steps were implemented to produce copper indium gallium diselenide (CIGS) solar cells. Composition and structure correlated to performance.



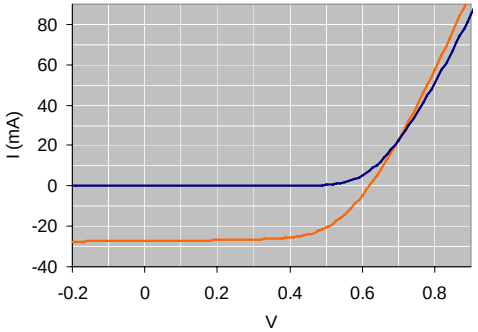


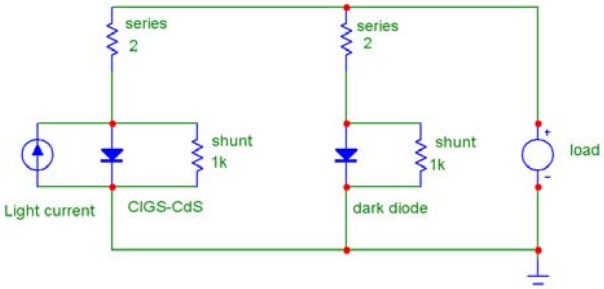
Complete solar-cell fabrication





Structural and chemical analyses





Photovoltaic performance and device model

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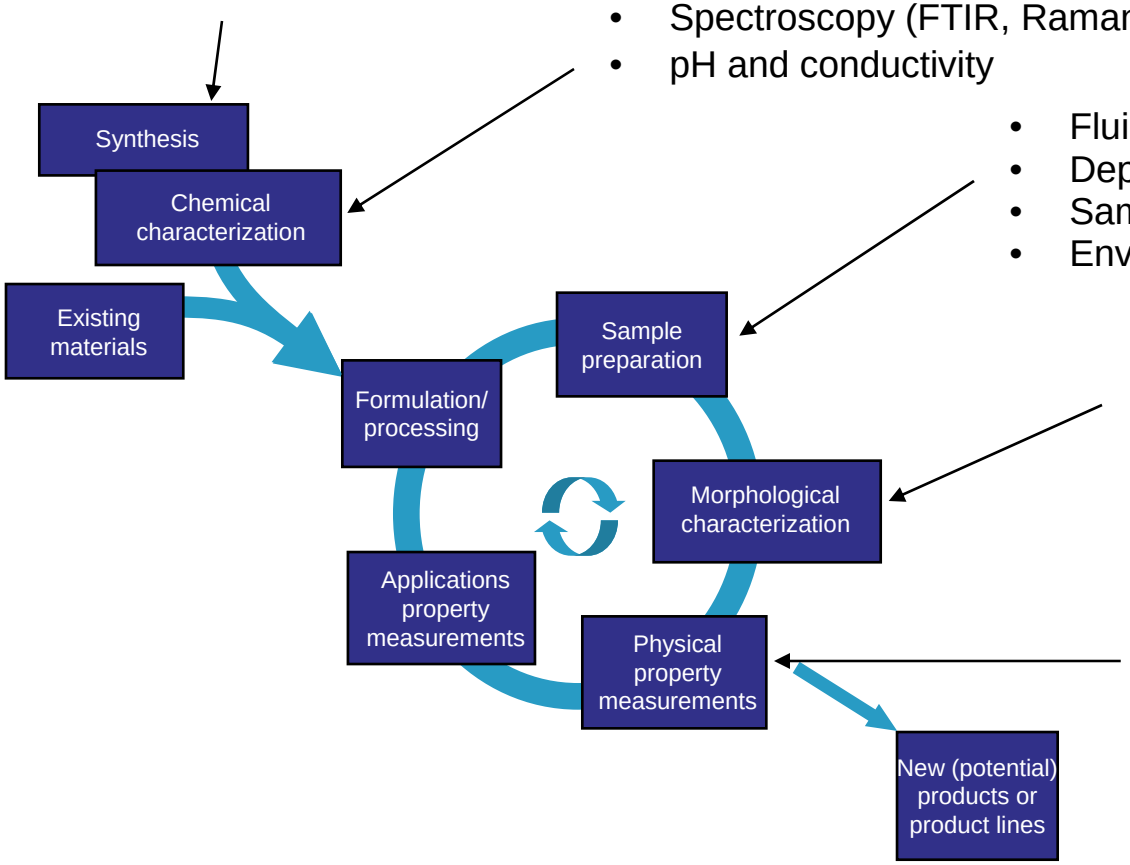
Symyx has High Throughput Capabilities for Product Development

- Batch and semicontinuous synthesis at controlled temperatures and pressures
- Mixing

- Chromatography (SEC, HPLC, TLC, GC)
- Spectroscopy (FTIR, Raman, UV-VIS)
- pH and conductivity

- Fluid handling, molding, coating
- Deposition on relevant substrates
- Sample processing
- Environmental perturbations



- Particle size/stability
- Turbidity
- Small angle X-ray scattering
- Viscometry and rheometry
- Modulus
- Failure
- Adhesion
- Tack
- Friction
- Chemical resistance
- Permeability
- Surface energy
- Optical measurements, *and more…*

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