DESIGN AND PROCESS CONTROL ISSUES IN NANOMANUFACTURING

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1. PRIMARY QUESTION

How can we achieve effective design and control of product properties and assure acceptable end-use product performance?

- In manufacturing products designed for specific end-use applications (e.g., polymer nano-composites) or
- In the production of self-assembled nanomaterials with desirable structural characteristics?
2. MACRO-SCALE NANOMANUFACTURING

Key defining characteristics

- Incorporating nano-particles into the matrix of other materials
- To achieve “tailored” material characteristics

Prototypical example: Polymer nano-composites
Polymer nano-composites

- Characteristics
  - Nano-filler component
    - cellulosic nano-fibers,
    - carbon nanotubes,
    - metal oxide nanotubes,
    - nanoclays
  - used to reinforce host polymer matrix
  - Show enhanced end-use properties compared to their conventional counterpart
Polymer nano-composites

Primary Issues: Property design
- Factors affecting evolution of material properties
- Optimal selection to achieve objectives
- Example: in polymer-clay nanocomposites, key issue is DISPERSION
Polymer nano-composites

- Primary Issues: Manufacturing process design and operation
  - optimum combination of operating conditions, equipment design parameters, and organoclay and coupling agent chemistry (i.e. integrated product and process design) required to facilitate dispersion and exfoliation
Polymer nano-composites

- Primary Issues: Process Control
  - maintaining optimum conditions in the manufacturing process, in the face of all sorts of variability, and ensuring that the design targets are met consistently in the final product;
  - Incorporating customer feedback information (the ultimate measure of product performance in end-use)
Customer Feed-back

- Binary in nature
  - Acceptable = 1;
  - Unacceptable = 0;

- Related to directly to end use property, but
  - Determined during manufacturing
  - Can be inferred *ahead of time* from process and product quality data!
Current State

- Edisonian approach to property design
- Little or no consideration for systematic integration of product design, process design and control;
- Control limited to “process operating variables”; no consideration for product quality and end-use attributes
Key Challenges

- Data Acquisition, Analysis & Information Processing
  - multi-scale, high-, low-, and medium frequency data analysis;
  - continuous, discrete and categorical data analysis;
  - image processing and analysis;

- Modeling
  - Nature and structure
  - Strategies for à-priori model development
  - Control-relevancy
  - Model update with operating data

- Control system structure, design, implementation
Possible Control Structure

Controller C3

Controller C2

Controller C1

Reactive Extrusion

‘q’, ‘w’ Pred.

‘q’ / ‘w’ Meas.

Customer Feedback

Initial Specs

\[ q^*, w^* \]

\[ y^* \]

\[ y \]

\[ u \]

Controls quality and end-use properties

Rejects unmeasured dist.

Translates customer feedback into set-points for ‘q’ and ‘w’
3. NANO-SCALE MANUFACTURING

- Key defining characteristics
  - Achieving desired product quality via explicit regulation of events at the *molecular* and *nanoscopic* length scales;
  - Manipulated variables adjusted during processing at *macroscopic* length scales (e.g. temperature, flows, applied potential between two electrodes, etc)
Illustration: Pb nanostructures on Cu(111)

Effect of film thickness

Different Pb nanostructures observed with increasing film thickness

Courtesy: Chatterjee and Vlachos

* Pb/Cu(111) structures
Nature (2001), 412, 875
Key Challenges

- On-line measurements?
  - AFM is off-line “after the fact”
  - Development of appropriate on-line analytical techniques for characterizing nano-structures?

- Observability
  - Inference of unavailable states from measurements

- Controllability
  - Insufficient MV’s, at the right scale
  - What modes to control actively, what to “force” by design to evolve naturally via self-assembly? And HOW?
Challenges in experimental control of nanostructures

- Identifying best synthesis protocol for obtaining desirable structures
- Phase diagram depends on several experimental variables
  - Temperature, film thickness, material properties, deposition rate, ...
  - Dependence not explicit or straightforward to quantify (in a control-relevant manner)
  - Multi-scale modeling useful for simulation (has its own challenges); inadequate for on-line control
- Experimental trial-and-error most common, but time consuming
Possible approach

- Optimal Control using Statistical Design of experiments
  - Quantize synthesis trajectory into finite “decision” variables
  - Use simulation and DoE to investigate and “quantify” effect of potential protocols on desired structure
  - Optimize, confirm, and implement
Illustration

Overview

Identify optimal synthesis protocol for
- Particle size = 6.8 nm
- Circular nanoparticles
- High-density arrays
- Hexagonal close packing
- Narrow distributions

Flowchart

- Identify key variables, select initial parameter range

- Response surface (RS) fit
  - Find minima in RS

- Optimum lies inside range?
  - Yes
  - Stop
  - No
    - Select new parameter range

- Parameter space sampled
- Response surface fit

- Minimize \( \min (\Theta) \)
  - Synthesis protocol
  - Fabrication Time
  - Target structure
Sample results

Desirable nanoparticle arrays obtained reliably

<table>
<thead>
<tr>
<th></th>
<th>Size (nm)</th>
<th>Aspect ratio</th>
<th>Spacing (nm)</th>
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</thead>
<tbody>
<tr>
<td>Best from iteration 2</td>
<td>6.5 ± 0.5</td>
<td>0.82</td>
<td>11.5</td>
</tr>
<tr>
<td>Optimal</td>
<td>6.78 ± 0.4</td>
<td>0.86</td>
<td>11.6</td>
</tr>
<tr>
<td>Target</td>
<td>6.8</td>
<td>1</td>
<td>Minimum</td>
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Summary and Conclusions

- Two aspects of nano-manufacturing considered
  - Incorporation of nano-particles
  - Multi-scale manipulations of nano-structures
- Current state of process Design and Control summarized
- Key Challenges noted
  - On-line measurements
  - Observability
  - Controllability
  - Integration of Design and Control (multi-scale manipulation; design to force evolution via self-assembly)