Microencapsulation Studies for Mass Production of IFE Targets

B. Vermillion, G. Besenbruch, L. Brown, D. Goodin, B. McQuillan, M. Takagi, T.Woo

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Goals of Experimental Study

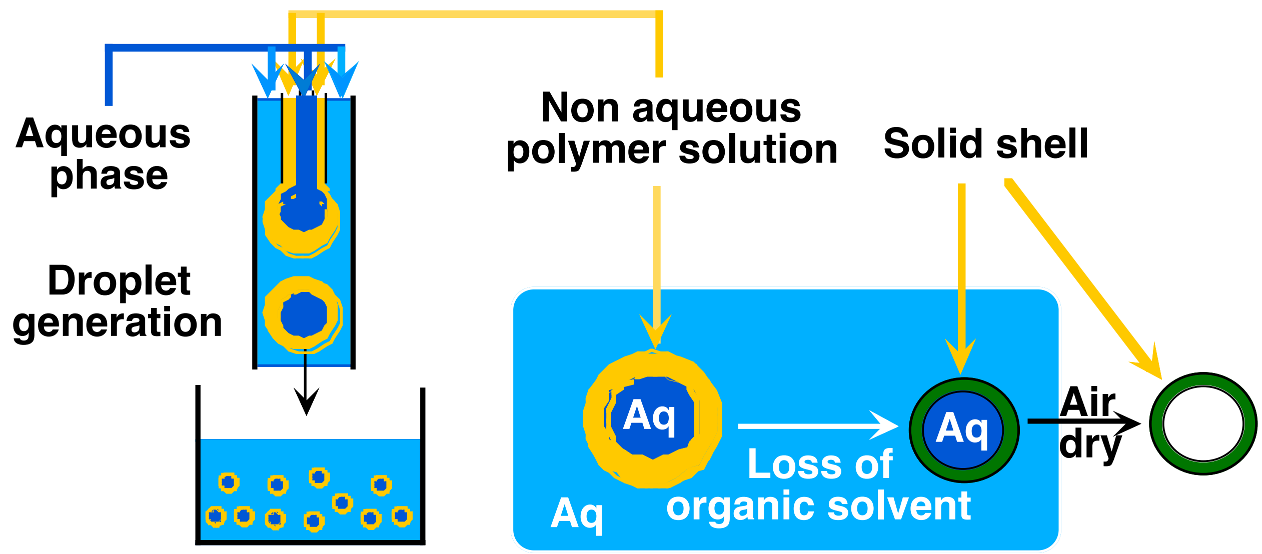
Overall

- Examine scale-up opportunities utilizing chemical engineering principals and apparatus
- Study methods to reduce costs associated with mass production

Specific

- Produce 4.6 mm/250 micron wall polystyrene targets
- Produce a stock of targets for additional coating studies
- Design and test apparatus for mass production of targets

Microencapsulation utilizes a triple orifice generator



- Three Phases
  - Inner aqueous phase (DI water)
  - Polymer phase (polystyrene in solution with fluorobenzene)
  - Outer aqueous phase (poly acrylic acid in DI water)

GENERAL ATOMICS
AND AFFILIATED COMPANIES

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Mass Production of IFE Targets differs from Lab Scale Production

- **Desire to scale-up production of polymer targets to at least 500,000/day**
  - **Liquid ratio assumptions**

    Ratio = Volume of solution plus targets/volume of targets

    Lab scale 200 : Production scale assumes 10
    - **Need for recycle: >3,000 L of outer aqueous phase per 100,000 target production run**
    - **Methods to remove contaminants**
  - **Parametric study to determine flowrates**
    - **Operating in a different regime for IFE target production**
- **Optimizing cure times**
  - **Surface defects, low mode**
    - **Controlling the Marangoni effect**
    - **Rate of fluorobenzene removal**

$$M = \frac{\left(\frac{d\sigma}{dc}\right)\Delta C\, L}{\mu D}$$

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Target Specification influence scale-up decisions

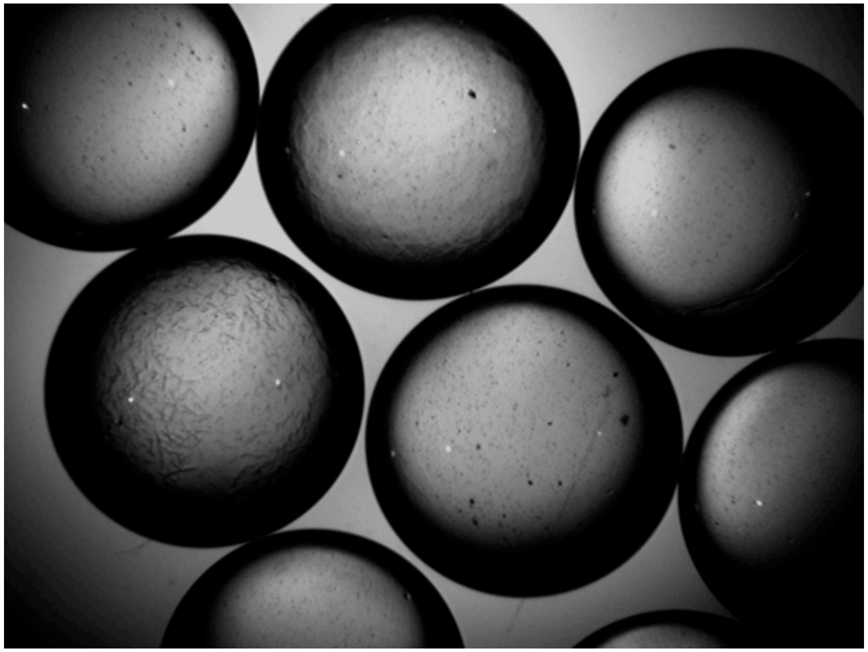
- Possible specifications for larger targets affect scale-up design and cost
  - OD: 4600 +/- 5%

  Over and Under: Acceptable Yield?
  - Wall: 250 +/- 10%
  - OOR: <1% of radius

  If one assumes OOR scales with R^3:

$$\frac{3mm}{0.36\%} = \frac{4.6mm}{0.86\%}$$

  - NC: < 1% to 5% of wall thickness
  - Surface Finish: 20-200 nm RMS



Polystyrene targets: 3 mm diameter/30 micron walls

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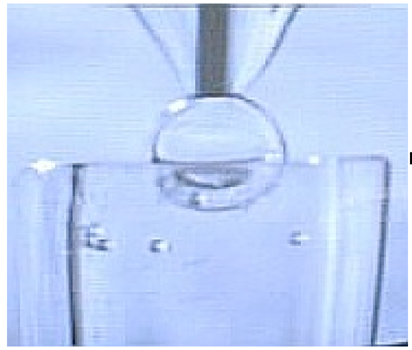
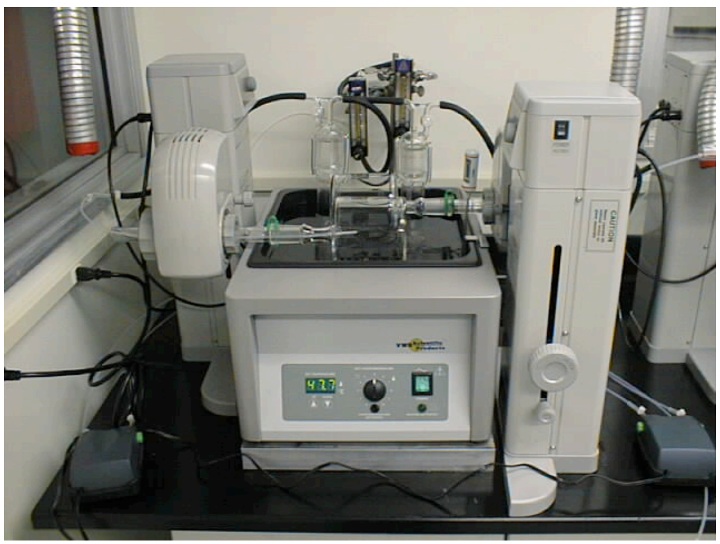
Exploring Scale-Up Opportunities



Droplet Generation

Laboratory Scale

Mass Production



Rotary Contactor

Prototype Gas Agitated Contactor

IFE Shell-Making Laboratory is in Operation

Process Conditions

Run #	O1 Composition and Flowrate	W1 Composition and Flowrate	W2 Composition and Flowrate	Drop Rate, shells/s	Inner Needle, mm	Outer Needle, mm	Filtered Solutions	Notes
1	8.0 wt% PS/FB, 20 cc/h	DI, 20 cc/h	0.3 wt% PVA, 2400 cc/h	1	0.2 ID 0.51 OD	0.7 ID	no	Shakedown Run
2	8.0 wt% PS/FB, 20-40 cc/h	DI, 20-40 cc/h	0.3 wt% PVA, 2400-3600 cc/h	1 to 6	0.2 ID 0.51 OD	0.7 ID	no	First Run
3	18 wt% PS/FB, 14 cc/h	DI, 40 cc/h	0.05 wt% PAA, 3750 cc/h	1.7	0.2 ID 0.51 OD	0.7 ID	no	Second Run
4	18 wt% PS/FB, 17 cc/h	DI, 48 cc/h	0.05 wt% PAA, 2400 cc/h	1	0.5 ID 0.8 OD	1.2 ID 1.5 OD	yes	Larger shells with filtered solutions
5	18 wt% PS/FB, 17 cc/h	DI, 48 cc/h	0.05 wt% PAA, 2400 cc/h	1	0.5 ID 0.8 OD	1.2 ID 1.5 OD	yes	GAC and DI water wash

Results

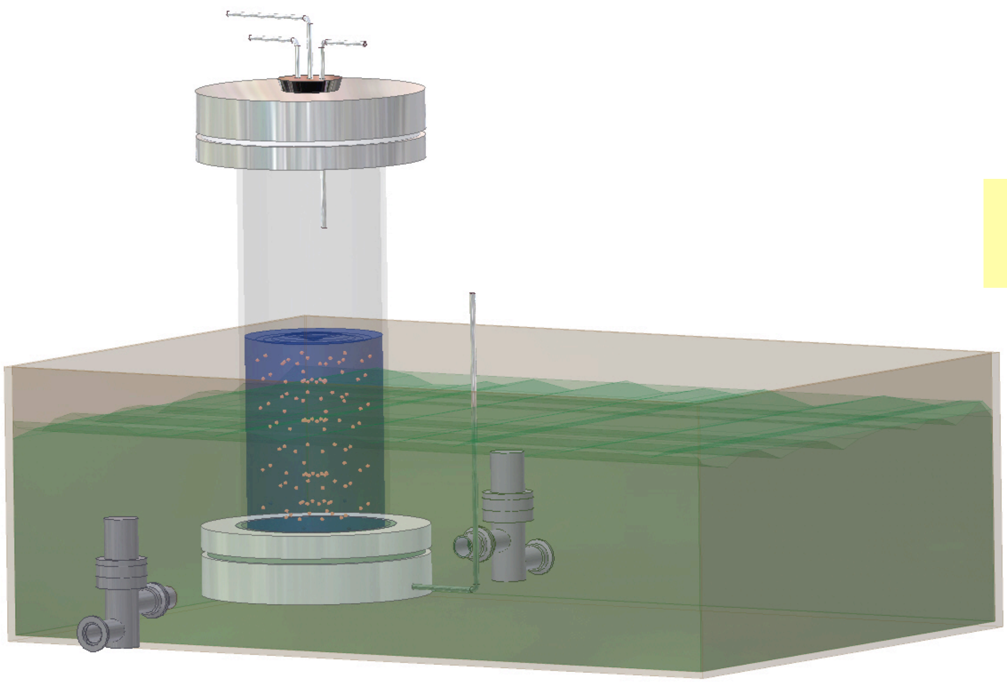
Specifications	Run 3	Run 4
Estimated Diameter	2,300 micron	3,003 micron
Estimated Wall Thickness	24 micron	30 micron
Diameter +/- 5%	2,387 micron, StDev 24	3,013 micron, StDev 8.2
Wall +/- 10%	23 micron, StDev 0.9	32 micron, StDev 1.3
OOR, < 1.0% of radius	0.44%	0.36%
NC, <1% to 5%	NA	1.4 +/- 1.6 %
Number of shells	~ 2,000	~1,000

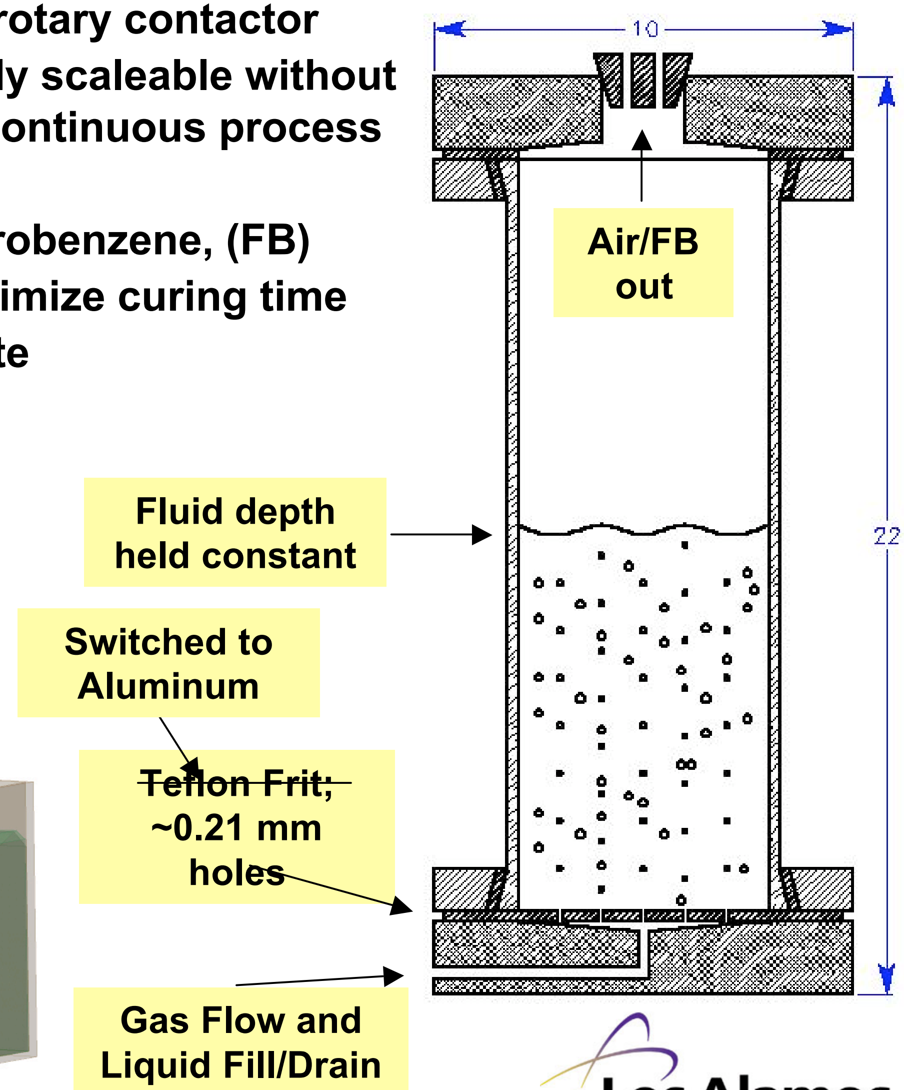
- **Run 5 to be characterized**

Microencapsulation Lab

Potential new method for curing, extractions, and nucleation

- Gas Agitated Contactor to possibly replace rotary contactor
- More suitable for mass production; eminently scaleable without repeating parametric studies; adaptable to continuous process
- Functional Attributes
  - Bubbles agitate targets and remove fluorobenzene, (FB)
  - Controlled FB to air concentration to optimize curing time
- Status: Design complete; initial run complete





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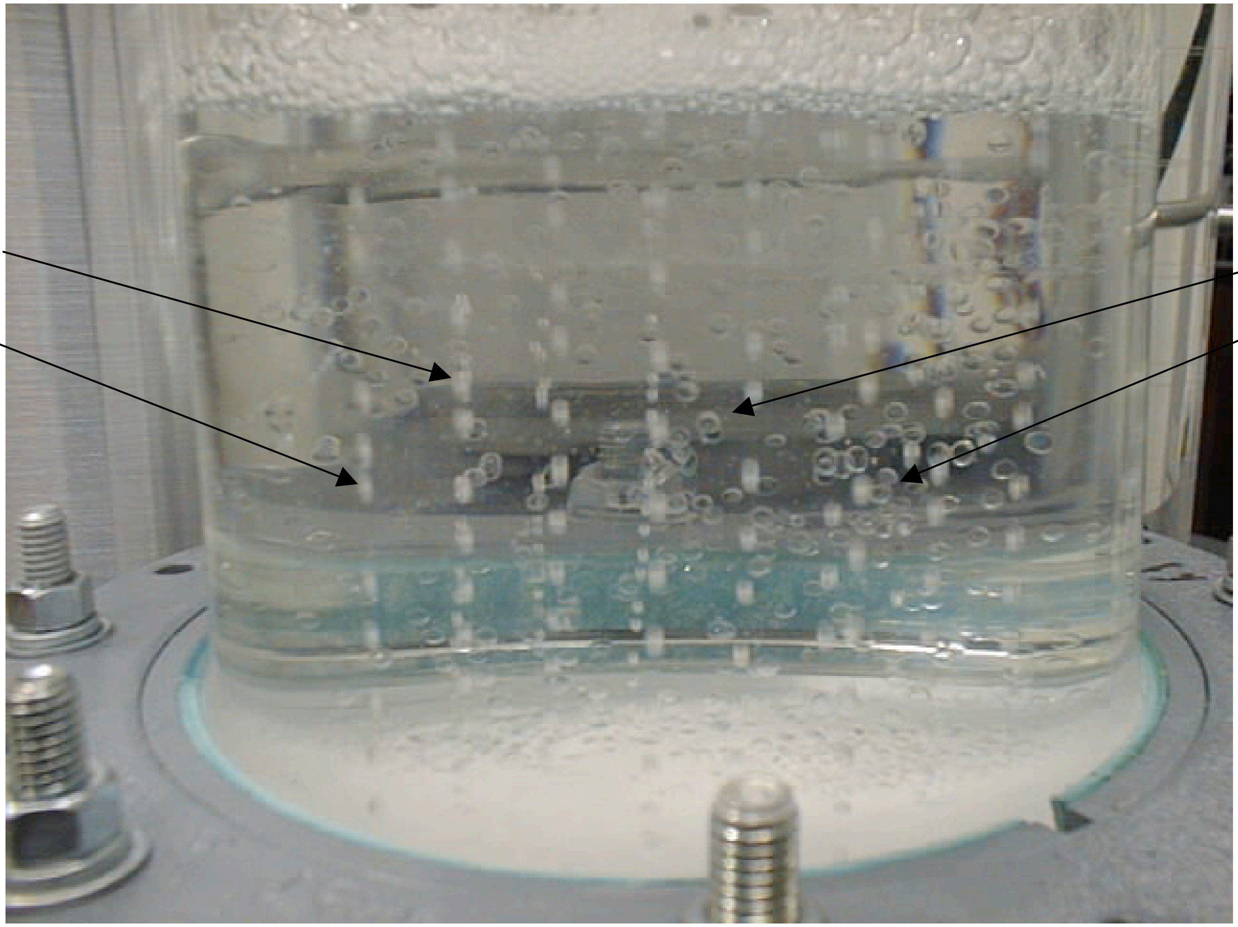
Gas Agitated Contactor Provides Uniform Mixing

Bubble Streams

PS targets



Gas Agitated Contactor initial run: 3 mm PS targets in 0.05 wt% PAA solution

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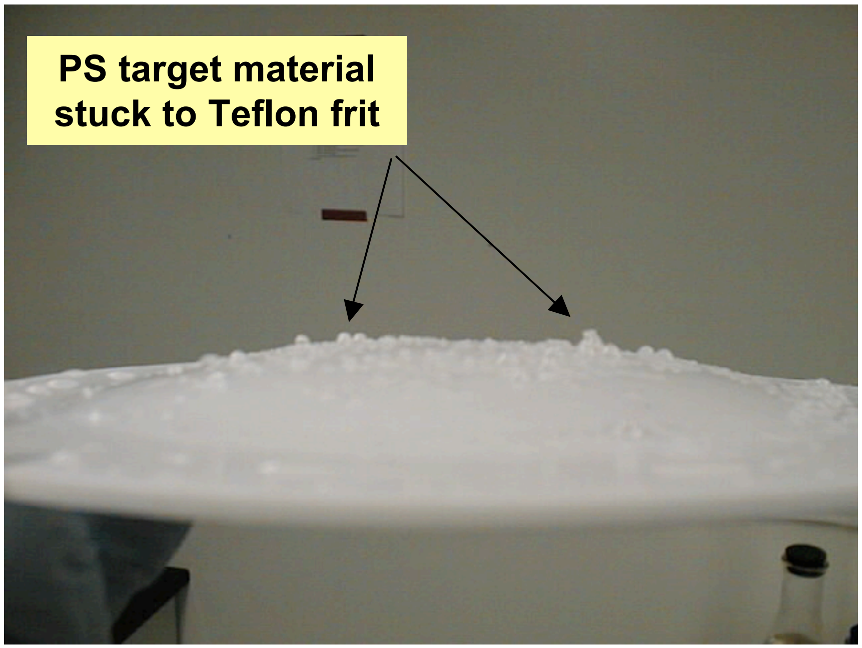
Gas Agitated Contactor Requires Foam Minimization

- Three washing solutions tested for foaming
  - 0.05 wt% PAA: Foaming inconsequential
  - 0.3 wt% PVA: Foaming moderate, drops of IPA broke apart bubbles
  - 3.0 wt% PVA: Foaming excessive, IPA drops did not alleviate
- Therefore, desire to identify a new washing solution with dual roles:
  - Ability to wash PAA off targets without foaming
  - Can be utilized in a continuous process without excessive cost
  - Several other washing solutions have been suggested:
    - DI Water
    - Isopropanol
    - Ethylene Glycol
    - Sodium Hydroxide
    - Acetic Acid

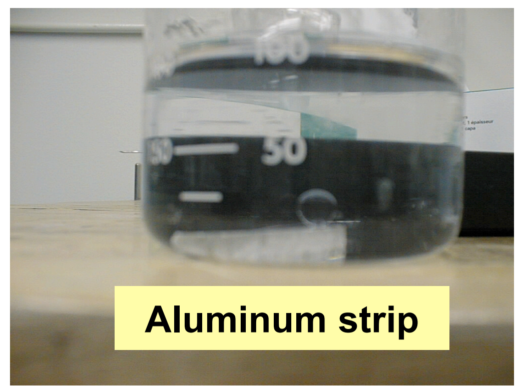
Fluorobenzene/polystyrene interacts with Teflon frit

- Fluorobenzene wets Teflon at a < 90 degree contact angle



- Altering frit material of construction to aluminum

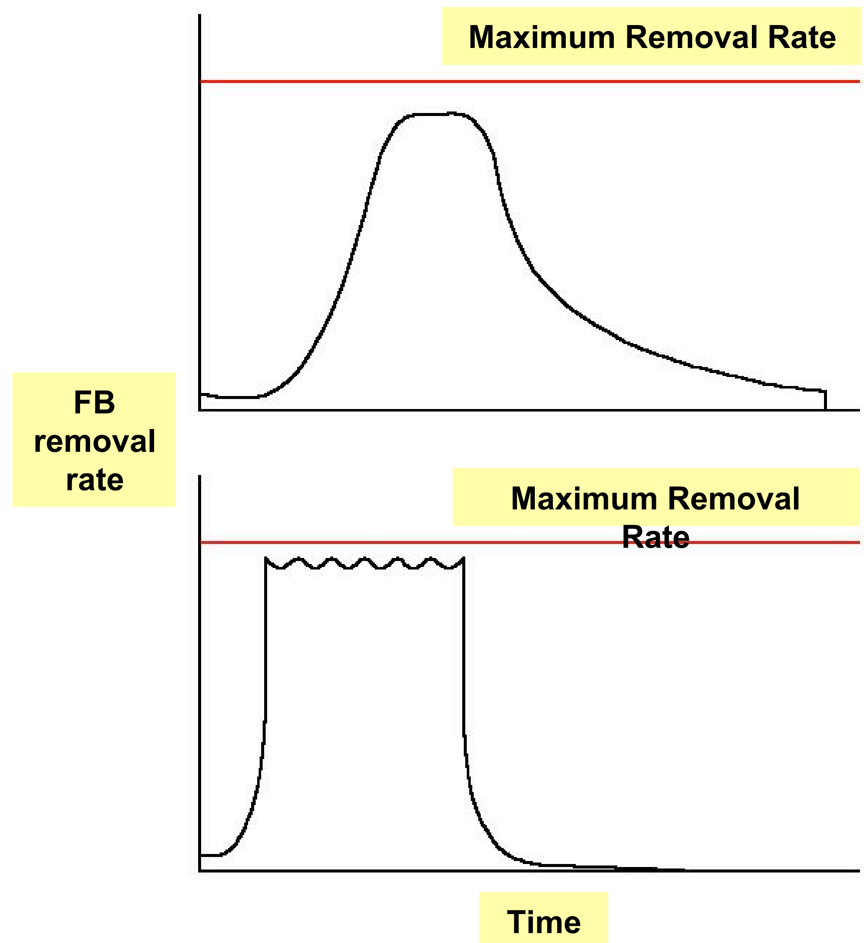
FB in 0.05 wt% PAA, contact angle < 90



FB in 0.05 wt% PAA contact angle ~180

Microencapsulation work near term

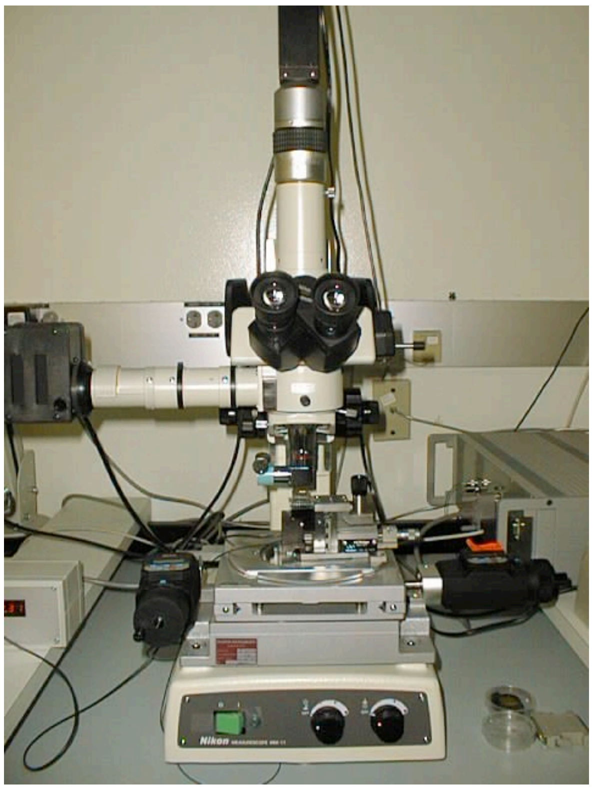
- Redesign of droplet generator
  - 5 mm wet target diameters
  - Redesign glass needle as mass production will require greater reproducibility
- Flowrates for larger targets; Double the flowrate, double the targets?
- Examine alternative wash solutions
- Heating of targets to decrease drying time
- Explore solvent removal rates to optimize cure time
  - Flame ionization detector to characterize FB concentration in purge stream



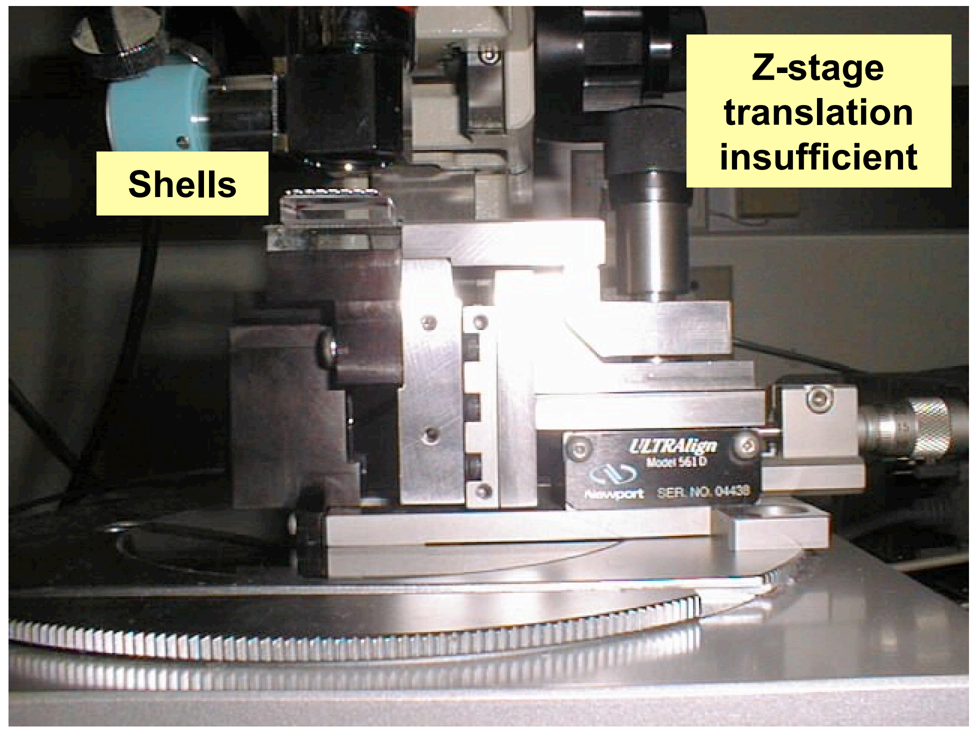
Conceptual optimization of curing time

ICF characterization techniques/equipment are insufficient for IFE targets

- Desired characterizations may determine which system is best for measurements
  - Inferometry: Stage size, optical focus
  - New automated optical system: layered targets, thick target walls



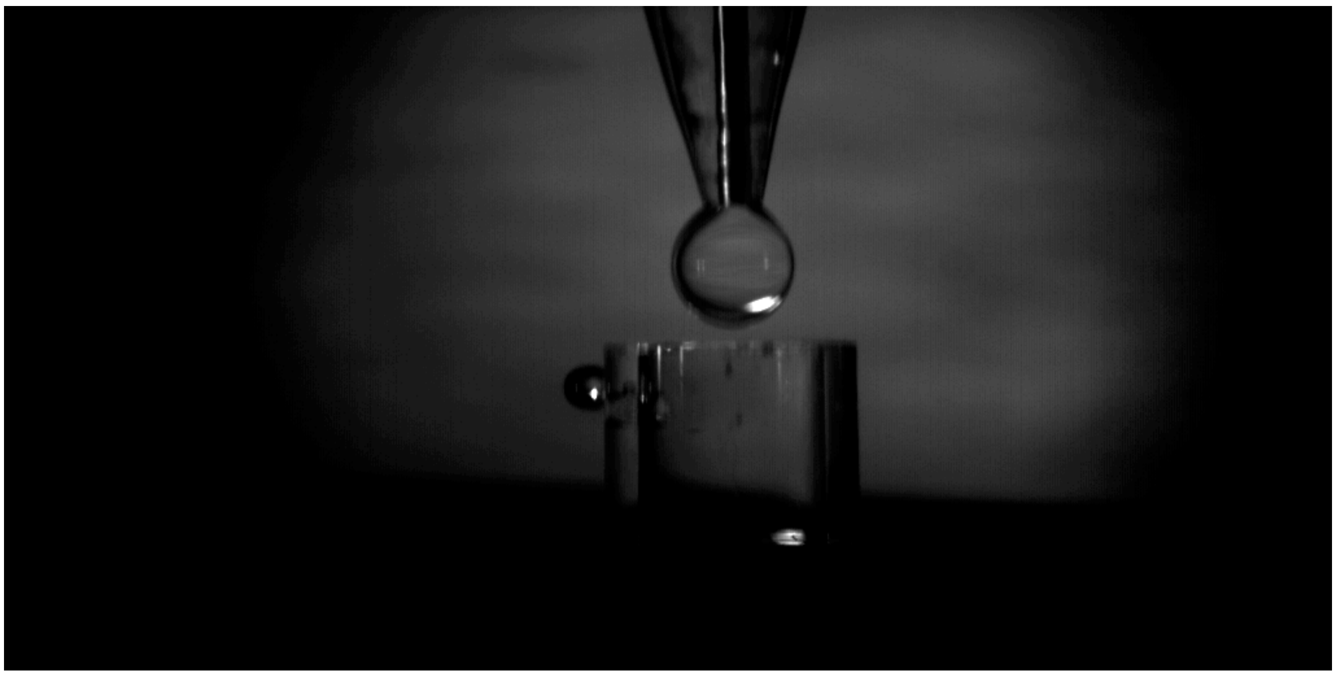
Laser Inferometry Characterization



Laser Inferometry System Close-up

More scale-up development is needed

- Examine methods for earlier characterization of shells
  - FID, process control
  - Optical, line scan cameras for target
- Explore pulsing as an addition to droplet generator
  - Increase droplet generation to >200 Hz; Reduce debris in process stream



2 mm PS target, ~1,000 frames per second

$$Time = \frac{1}{6} \cdot \frac{L^2}{D_{AB}}$$

- Explore other methods for reducing drying time
  - Nucleation (in 100 % ethanol), Forced convection

Microencapsulation Studies Recap

Examine scale-up opportunities utilizing chemical engineering principals and apparatus; study methods to reduce costs associate with mass production

- Gas Agitated Contactor design and implementation
- Study of alternative washing solutions including DI water
- Further literature review and theoretical work

Produce 4.6 mm/250 micron wall polystyrene targets

- Experiments are approaching larger diameters, already formed 3 mm targets, limit of current droplet generator design
- Work still needed to determine process flowrates for larger targets and greater production rates

Produce a stock of targets for additional coating studies

- Targets are being produced now which can be utilized for further studies