Novel Foam Encapsulation Materials and Processes

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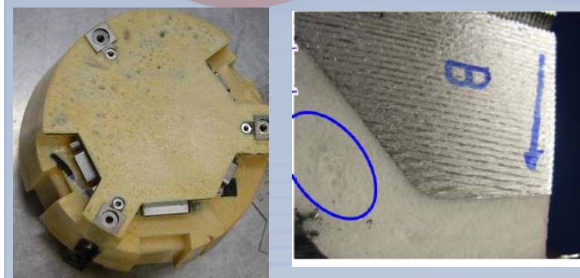
Problem Description and Goals of Project



Foams often have density gradients or inhomogeneous cell sizes

Overfilling is necessary in this geometry to eliminate large void

- Many weapons components (e.g. firing sets) are encapsulated with blown foams
- Foam is a strong lightweight material – good compromise between conflicting needs of structural stability and electronic function
- Current foaming processes can lead to unacceptable voids, property variations, cracking, slipped schedules – Long-standing issue
- Predicting the process is not currently possible – Material is polymerizing, multiphase with changing microstructure.

Goals:

- Produce uniform encapsulant consistently and improve processability
- Eliminate metering issues/voids
- Lower residual stresses, exotherm to protect electronics
- Maintain desired properties: lightweight, strong, no delamination/cracking, ease of removal

Approach

- Create a real understanding of the variables that influence the final foam material properties
  - Chemistry, surfactants, additives
  - Processing variables
  - Use this understanding to develop predictive computational tools
- Use a chemical rather than physical blowing agent (better control)
- Develop a WR foam that is created outside of the part and then pressure fill
  - Advantages:
    - Flexible conditions in which to make uniform cells (high P or T)
    - More uniform density in final product
    - Foam QA easier
    - Pressure filling expected to lead to fewer voids
  - Challenge: Foam must be super stable
- Develop a state-of-the-art computational capability to use early in the design process to allow optimization of hardware (gates, vents) and processing parameters

The Multidisciplinary Expertise Available at Sandia is Required

Team consists of polymer chemists, computational modelers, rheologists, process engineers, and specialists in experimental hardware development and techniques. *Breadth and depth of expertise only available here.*

Challenges:

– New chemistry
  - Stacked reactions to allow creation of viscous foam suitable to injection and second a rigidization mechanism
  - Development of hybrid materials (coupling epoxies with polyurethanes, or coupling epoxies with free radical polymerization mechanisms)
  - Surfactants / nanoparticle use

– Use diagnostics in pre-injection stage (QA before ruin part!)

– Develop computational predictive capability to aid in design & optimization of process (sensitivity to processing parameters, vent and gate locations, minimization of residual stress)
  - Complex 3D flow with an advancing front
  - Extremely challenging to get material properties

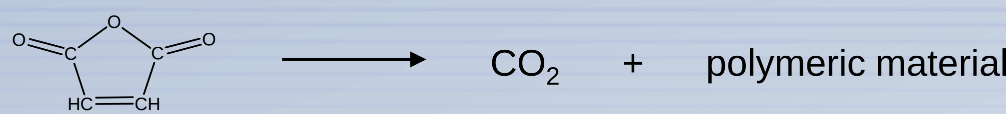
Many Chemical Foaming Processes Examined: Three Down-Selected

- Low functional isocyanate to yield CO_2 (limited as CO_2 production is coupled with MW increases and gelation)

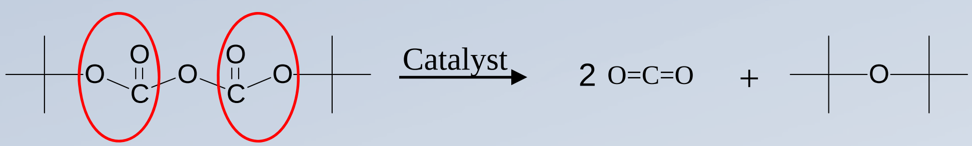
$$R{-}N{=}C{=}O \xrightarrow{H_2O} R{-}NH_2 + CO_2$$

"Stacked reactions" possible

- Maleic anhydride based foaming in non-reactive matrix



- Decomposition of butyl-dicarbonate (BOC) to generate CO_2



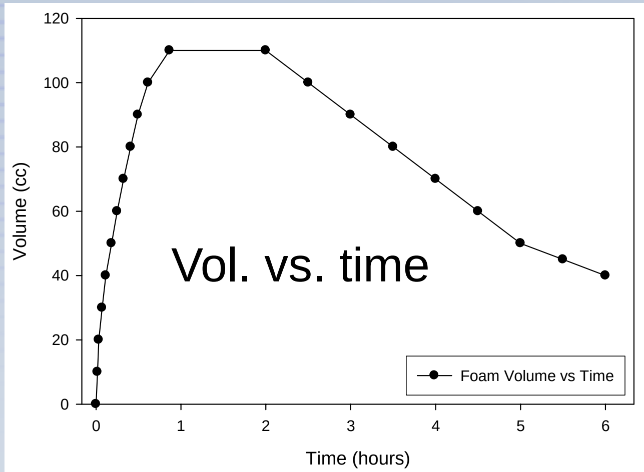
Patentable chemistry

Innovation: developed a non-curing foam for fundamental studies

Systematic Studies Of Constituent Influence On Foam Stability

Many factors will affect foam stability

- **Persistence of foam and stability under mechanical shear**
- **Initial screening studies to identify the key issues**
- **Base resin: Epon 161 foamed with BOC decomposition, non-curable**



Foaming reaction

Main matrix resin: Viscosity, chemical structure, blends

Surfactants, different classes

Fillers, additives, particles

Additional reactions increasing MW

- **Characterization of system viscosity, surface tension, rheology**
- **Persistence, vol. and cell structure vs time**
- **Stability under mechanical shear**

Example: Exploring Different Constituents

Increasing
resin viscosities

Base formulation
Epon 161 after 5 min

Poor
surfactant
after 5 min

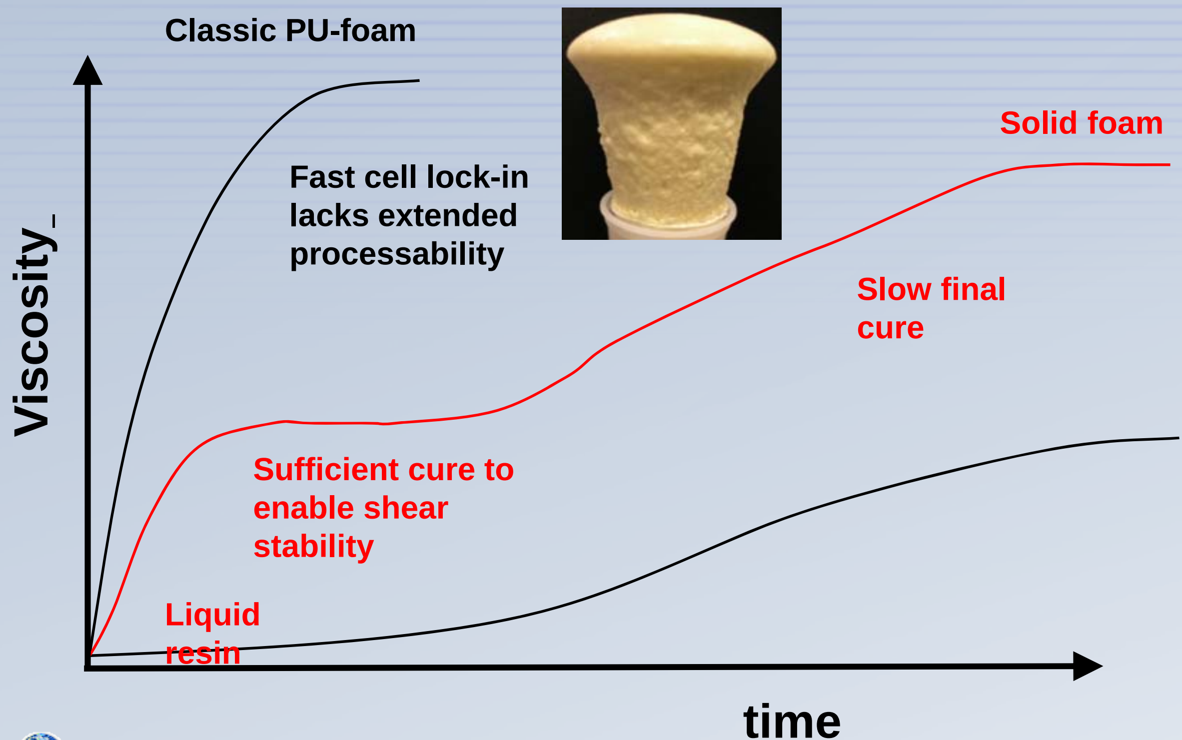
- **No direct correlation between resin viscosity and persistence**
- **Surfactants are very important for stability**
- **So far no significant stability enhancement observed using standard fillers (e.g. Cabosil) either**
- **Initial foam stability can be optimized via formulation adjustments but partial network formation yields much more robust foam**

Development of Shear-Stable Foam – Concept Proven

Strategy: Develop shear stable foam system that has sufficient polymer network support, but is still amenable to flow and processing (slow penetration of gaps)

Multiple approaches:

- **a) Tune-down polyurethane system, i.e. reduce initial network formation**
- **b) Enhance BOC blown system with sufficient initial cure to stabilize foam**
- **c) Use stacked cure, achieve initial stability of foam followed by slower matrix cure**



New hybrid system

Optimize: cure kinetics, crosslinking, gelation

Slow resin cure system

Extended processability, but cells collapse prior to cure

Shear Stability Tests

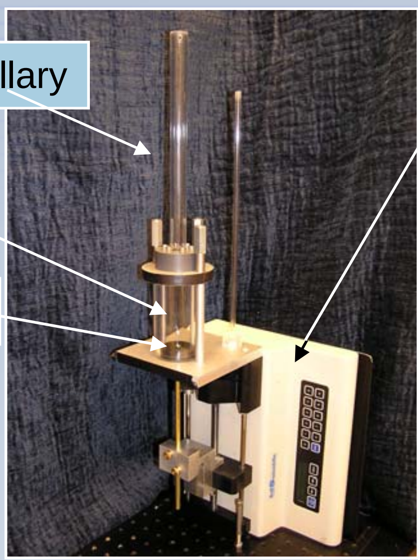
- Transparent capillary rheometer allows measurement of viscosity and slip while viewing microstructure
- Images taken for Particle Image Velocimetry and bubble size distribution
- Determine boundary condition for modeling
- Compare to conventional rheometry
- Similar apparatus being used to determine foam stability under shear conditions by measuring volume retention

syringe pump

capillary

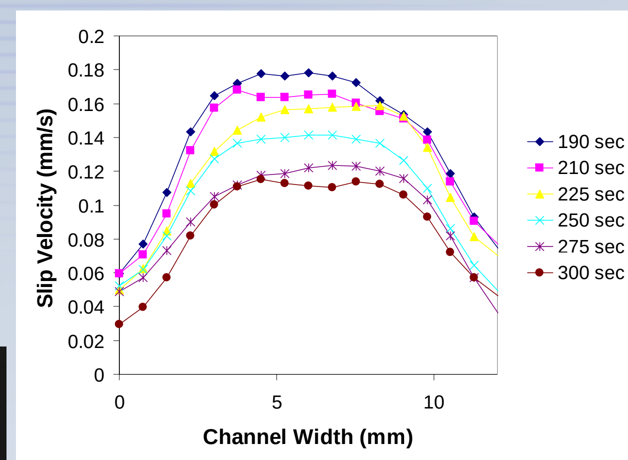
reservoir

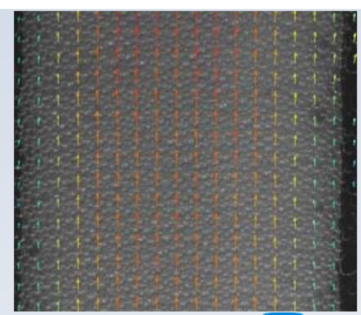
plunger



Capillary rheometer

PIV gives slip velocity at the wall





Example of Different Foam Stabilities

- Unstable foam, not optimized regarding surfactants and viscosity
- Stable foam with optimized surfactant but not yet crosslinked
- Improved system contains a physical network support via stacked reactions

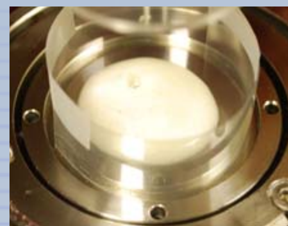
Unstable foam

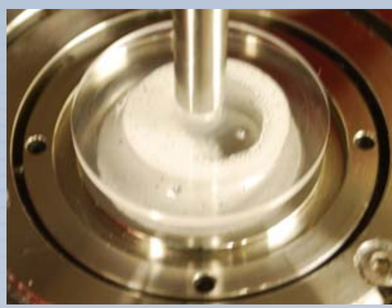
Stable foam for systematic foam stability experiments

Shear stable foam system
Polymer network support
Extended pliability for processing

Shear Stability Data

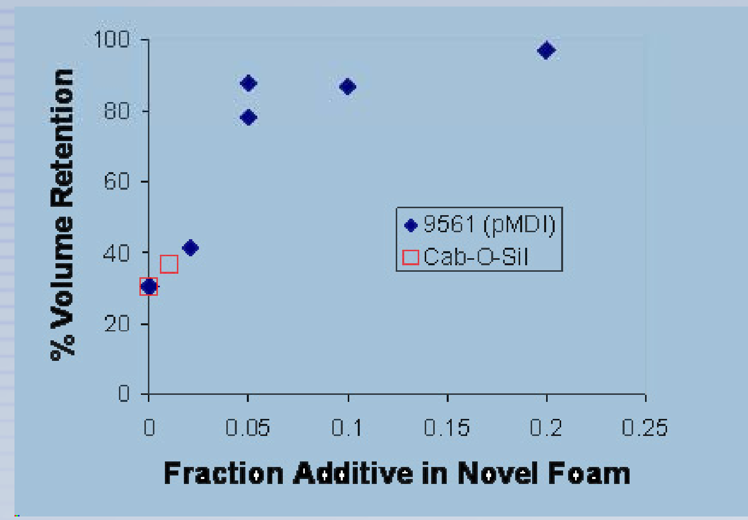
Foamed in place then compressed





After shearing, without cross-linking

With cross-linking

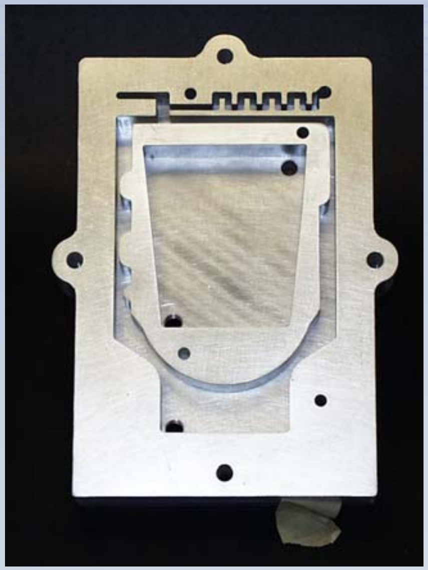


Conventional parallel plate rheometry with transparent plates
- Viscosity dependent on loading procedure
- Microstructure affects viscosity measurements
- Partially cross-linked foam more shear stable

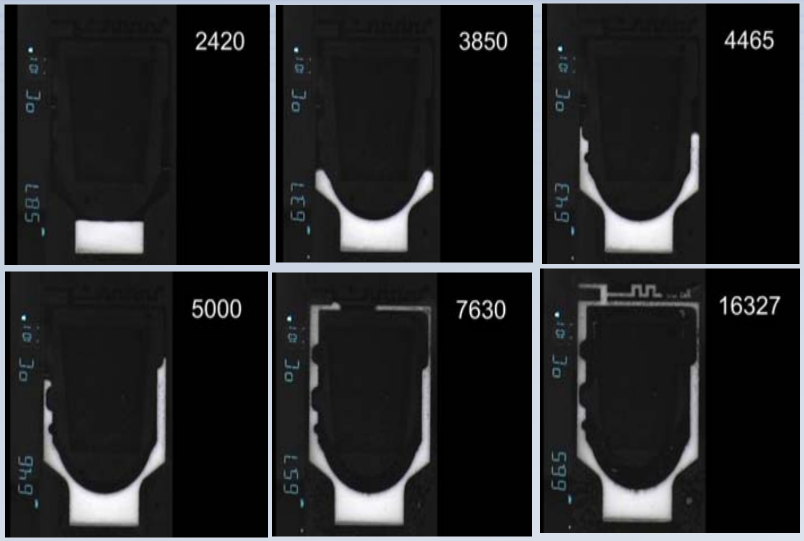
Capillary shear device
- Particulate additive shows poor volume retention
- Addition of a cross-linking agent shows good volume retention

Kansas City Mold Duplicated for Testing

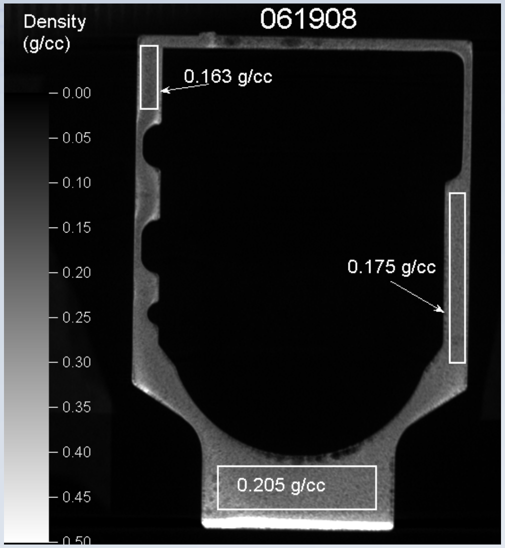
- KC-developed mold for QA of foams will be used to test down selected foams
- Foam outside the mold and injected using syringe pump apparatus similar to capillary rheometer
- Compare to foaming in situ to quantify difference in filling in small gaps
  - Determine density of final product using CT



Mold



Video recording of in situ fill



CT density

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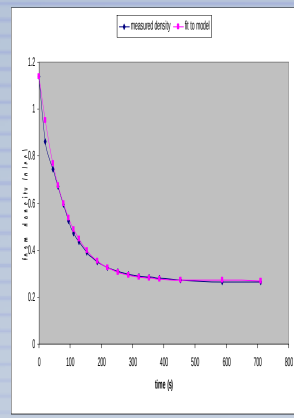
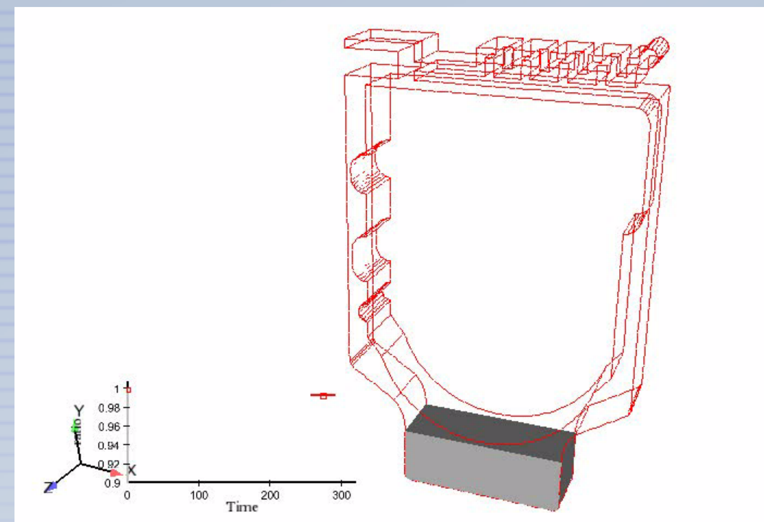
Finite Element/Level Set Model Improves Understanding of Foam Self-Expansion

$$\rho\frac{\partial \mathbf{v}}{\partial t} = -\rho\mathbf{v}\bullet\nabla\mathbf{v} - \nabla p + \mu_f\nabla^2\mathbf{v}$$

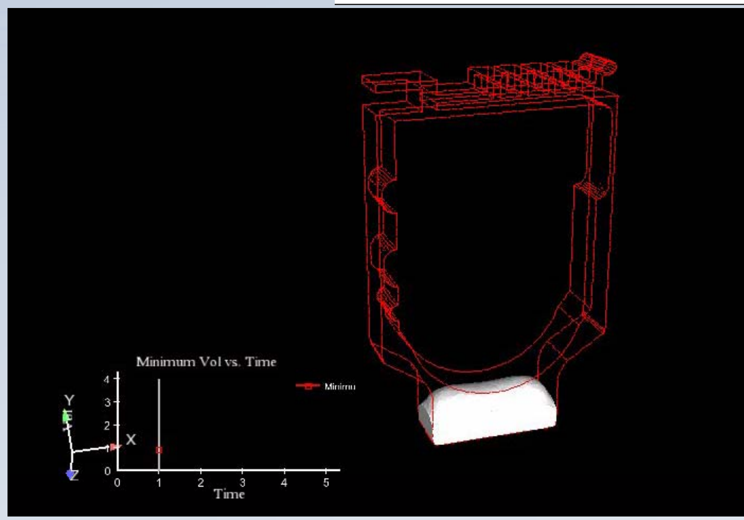
$$\nabla\bullet\mathbf{v} = -\frac{1}{\rho_f}\left(\frac{\partial\rho_f}{\partial t} + \mathbf{v}\bullet\nabla\rho_f\right)$$

$$\rho = \rho_{final} + (\rho_{initial} - \rho_{final})e^{-kt}$$



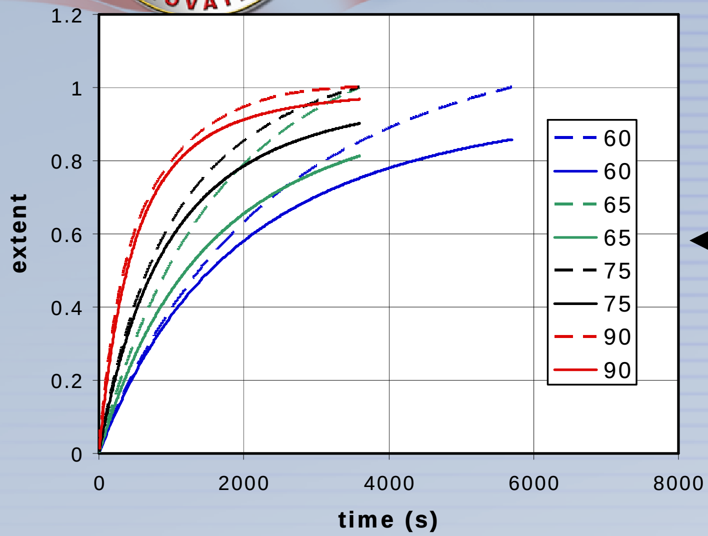


- Equations of motion are coupled to a time dependent density function fit from experimental data for EFAR20 foam
- Foam is assumed Newtonian
- Location of the free surface with time is determined from the zero of the level set equation
- Methodology can be applied to other foams



Pressure-driven flow of foam (L) and self-expansion (above)

New Kinetic and Viscosity Data Allow for Higher Fidelity Computational Model



- **DSC experiments give heat as a function of temperature for EFAR**
- **Extent of reaction fits somewhat better at higher temperatures**
- **Rate fit is more accurate implying heat evolution will be quantitative**

$$\frac{D\xi}{Dt} = k^i e^{\Delta E/RT}\left(1-\xi\right)^n$$

$$\Delta E = 11\frac{kcal}{mol}$$

$$k^i = 8.6x10^3\frac{1}{s}$$

$$n = 1.4$$

$$\Delta H_{rxn} = 223.75\frac{J}{g}$$

- **Raw viscosity data taken as a function of time and temperature**
- **Time and temperature shift give activation energy and temperature dependence of uncured system assuming an Arhennius form**

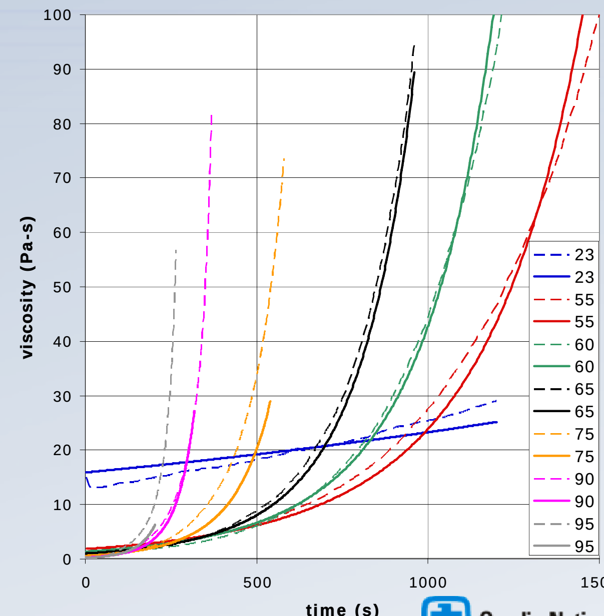
$$\eta_0 = \eta_{00}\exp(-\frac{E_a}{RT})$$

$$\eta = \eta_0(\frac{\xi_c - \xi}{\xi_c})^{-3.5}$$

$$\eta_{00} = 4.0x10^{-9}\,Pas$$

$$E_a = 13\frac{kcal}{mol}$$

$$\xi_c = 0.6$$



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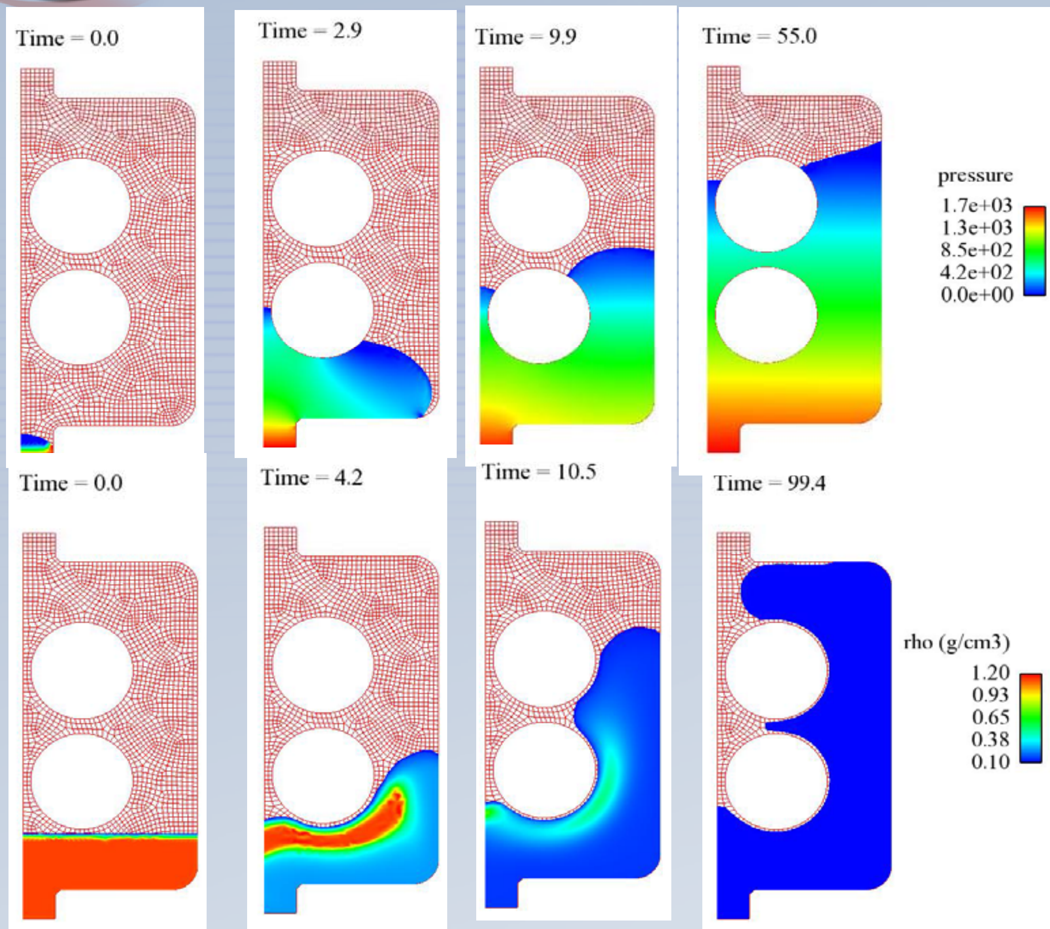
Pressure Driven Flow vs. Free Rise Foam



Pressure driven flow leaves smaller voids than free rising foam

Free rising foam has trouble entering interstitial spaces

Motivation to produce a new more stable foam outside the mold and pressure fill

Summary of Achievements in the First Year

- Developed patentable chemical foaming chemistry - TA
- Developed persistent non-curing foam for systematic evaluation of fundamental physics of foams
  - Initial testing of non-curing foam shows that surfactants very important
- Identified foam stability strategy using a stacked reaction scheme
- Developed foam rheology methodologies and shear apparatuses
  - Began testing candidates for shear stability
- Began development of computational model
- Development of methodology and collection of property measurements/boundary conditions for input to computational model

Significance

- Positive impacts to KC production expected:
  - **Encapsulation occurs at end of manufacturing process so expensive components are at risk if encapsulation is flawed.**
  - **Design/optimization of new encapsulation processes will be faster and easier with computational predictive capabilities.**
  - **These new materials and processes will help create nuclear weapons that are safer, more reliable, more easily transportable, and more resistant to aging since they will be better insulated in shock and vibration and will not exhibit residual stresses that can damage sensitive electronic components.**
  - **This work will improve yield, reduce cost, and minimize potential SFIs.**
  - **Quality metrics and diagnostics tools will be developed and transferred to KCP and Pantex.**
- Work is expected to put Sandia at the forefront of understanding foaming phenomena in general
  - **Ubiquitous in manufacturing**
  - **Defoaming strategies in processing nuclear and other wastes**
  - **Oil and gas drilling**