Fabrication of Hollow Resorcinol Formaldehyde Aerogel Spheres Using UV Photo-Initiators or Photo Activated Acids for (ICF) Experiments

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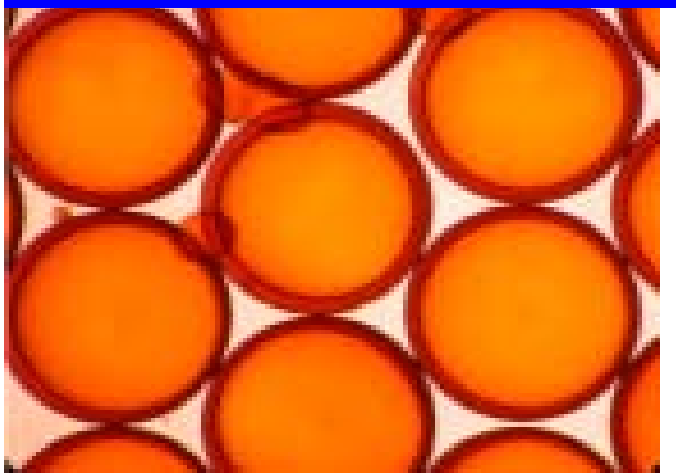
GENERAL ATOMICS

Resorcinol Formaldehyde (R/F) can be used for a variety of ICF experiments

ICF Experiments

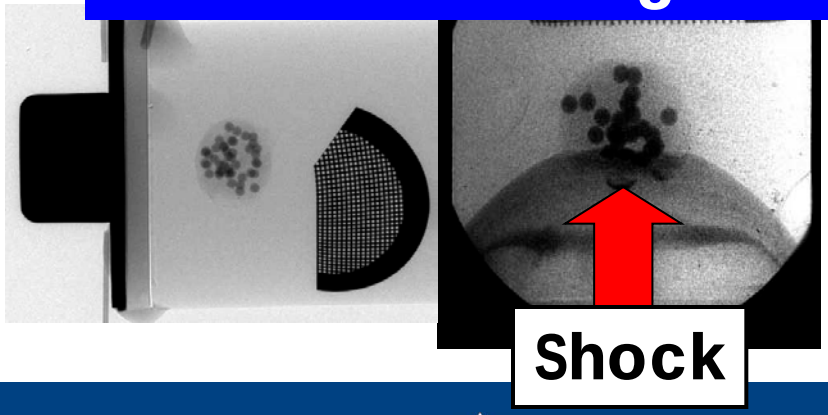
- Spherical Targets
  - Shells or Beads
- Astrophysical studies
  - (Jet/Shock)
- Cryogenic studies
- Perturbation studies

R/F Foam shells



200 μm Planar R/F film with a CH perturbation

Astroshock Target

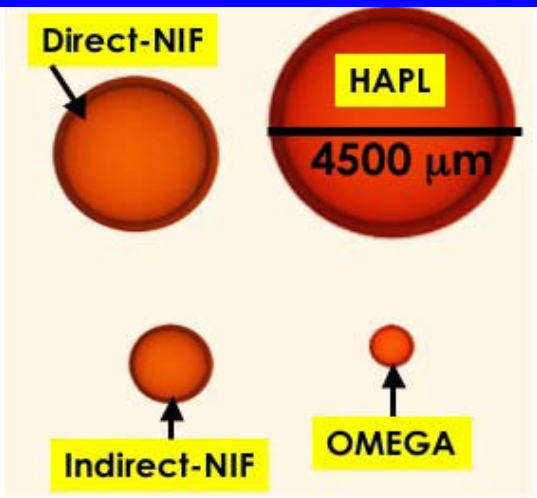


R/F aerogel capsules have been used for many ICF, IFE & experiments

- **Traditionally R/F is synthesized with 2 step polycondensation reaction***
  - (Base & Acid Catalyst)
- **Wall uniformity yield is low for sizes ≥ NIF (2 mm)**
  - (40-50%)**
- **Investigate alternative centering technique: Dielectrophoresis (DEP)[1-3]**
  - Centers the shells using electric fields
  - Centering traditional R/F is challenging with DEP

Why is centering traditional R/F using DEP more Challenging ???

Typical Types of R/F Shells



1.H. Pohl, Dielectrophoresis : the behavior of neutral matter in nonuniform electric fields, (1978)

2.T. Jones, Electromechanics of particles, (2005)

3.Bei, Z.M., et al., App. Phys. Letters, 93, 184101, (2008)

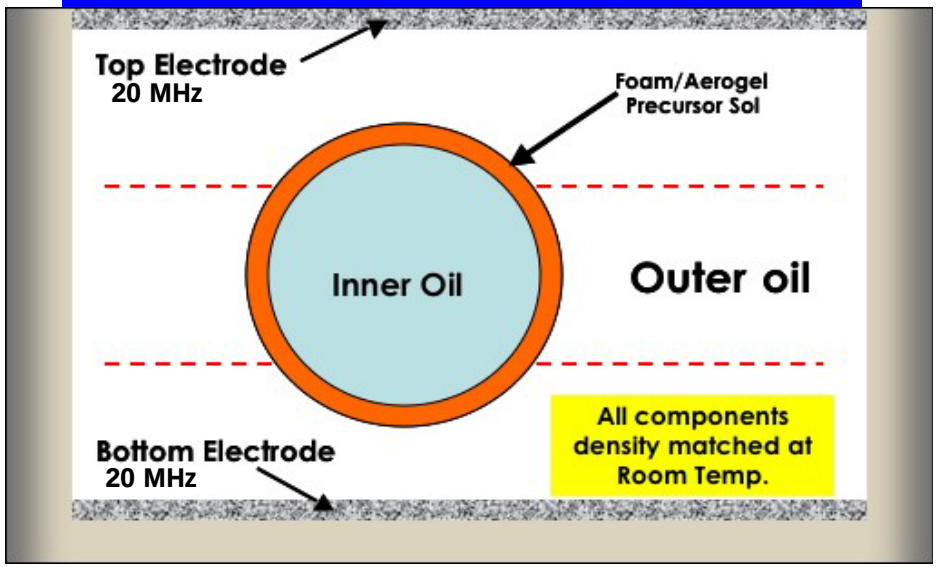
* Pekala, R.W.; et al. *J. Mat. Sci.*, 1989, *24*, 3221-3227.

** R.R. Paguio, *et al., Fusion Sci. Technol.* 59, 1, 199, (2011).

Traditional R/F is more challenging to center using the current DEP centering device due to it's conductivity

- **Traditional (R/F) conductivity ($5X10^{-2}$ S/m)**
  - Due to base & acid catalyst
  - Long cure time at room temperature
  - Current DEP centering device can center solutions $\leq 10^{-2}$ S/m

DEP Centering Device



1.Bei, Z.M., et al., App. Phys. Letters, 93, 184101, (2008)

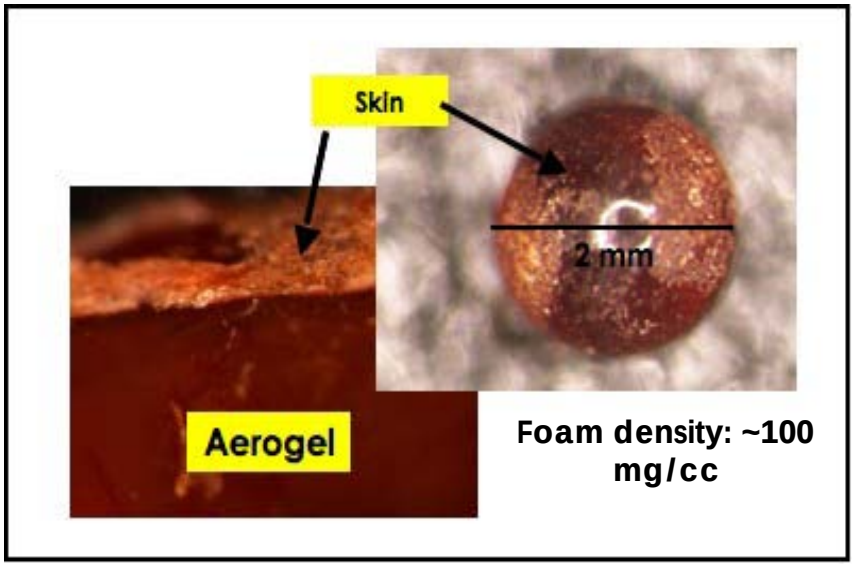
Solutions with higher conductivity are more challenging

* R.R. Paguio; et al., Mater. Res. Soc. Symp. Proc.,1306, 2011
^ K.M. Saito; et al., Mater. Res. Soc. Symp. Proc.,1306, 2011

DEP triggered the development of a novel method to synthesize R/F using UV photo-Initiators*^

3 types of modifications

- All 3 modifications gel at room temperature is 10-20 min.
- Proof of Principle: Fabricated a single bead by dropping modified precursor sol in oil & cured with UV
- One of the three modifications had the desired conductivity for the current DEP centering device
- Rough skin on the beads



- **Working to improve DEP device for centering experiments**
- **In Parallel: UV initiated R/F was used to fabricate shells using the traditional droplet generator method**

DEP Presentation by G. Randall; et al., Wed. PM-1:25PM

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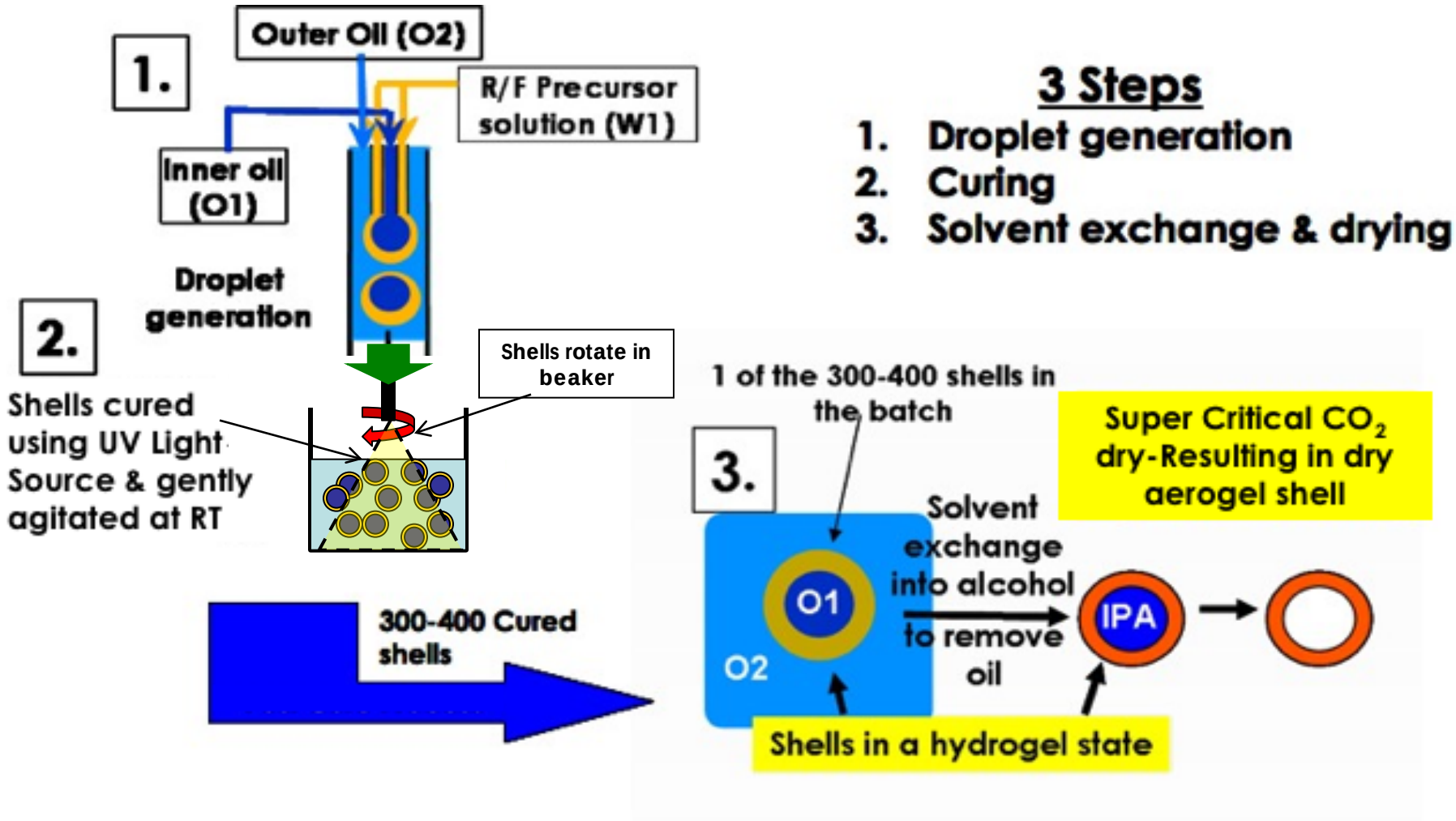
^ K.M. Saito; et al., Mater. Res. Soc. Symp. Proc.,1306, 2011

Droplet Generator used to fabricate hollow spheres using UV Initiated R/F & UV Photo acids



Traditional R/F synthesis uses a base catalyst to form primary particles that are combined to form the aerogel network using an acid catalyst

2 step poly condensation reaction

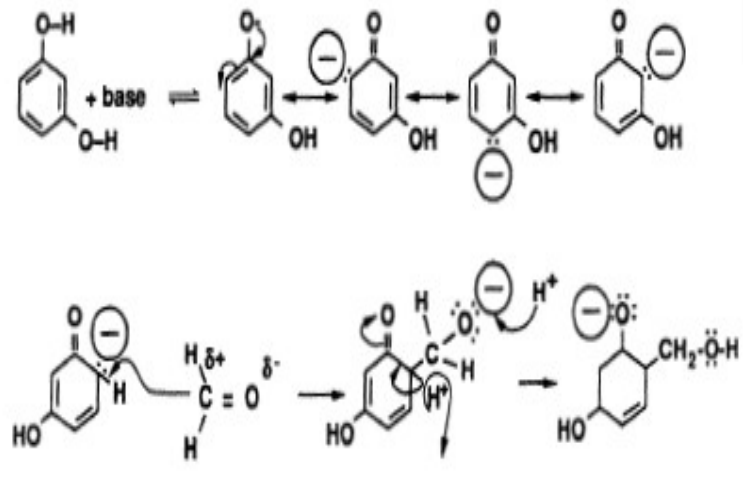
A: Base Catalysis

- Activation of Resorcinol ring
- Formation of hydroxymethyl adducts (primary particle)

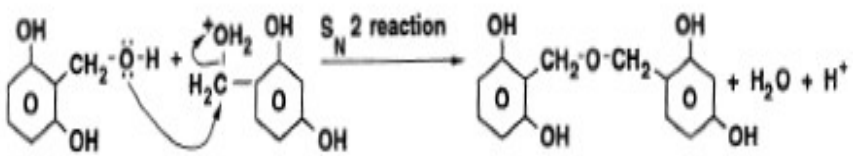
B: Acid Catalysis

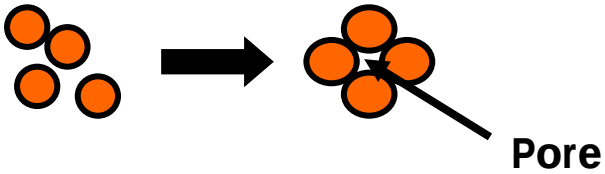
- Primary Particles form methylene ether bridges (pore formation)
- Makes gel stronger by increasing cross link density

A



B





* Pekala, R.W.; et al. *J. Mat. Sci.*, 1989, *24*, 3221-3227.

Modifications for synthesizing R/F using UV-Free radical photoinitators & photo acid generators (PAG)

1. UV initiator replacing only acid catalyst (Mod 1)

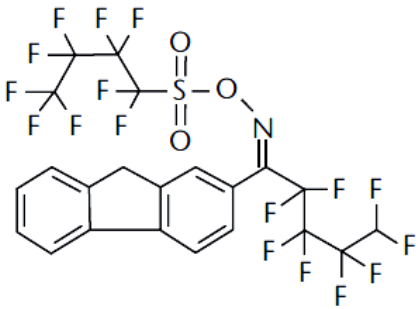
- Used normal base catalysis
  - Forms primary particles

Mod 1A: UV initiator is used to connect primary particles to form aerogel

Mod 1B: PAG is used to connect primary particles to form aerogel

- Conductivity too high For DEP

CGI 1907: Photo Acid Generator(PAG)

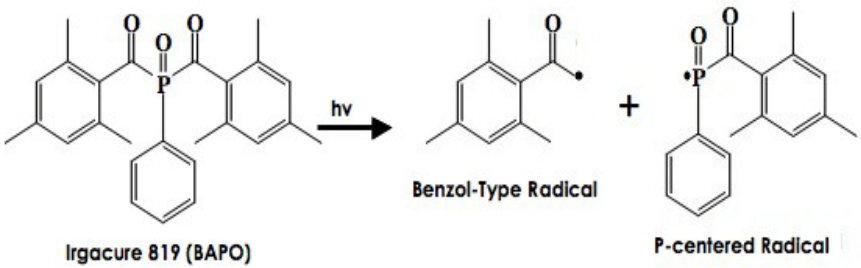


Forms Nonaflic Acid when exposed with UV light

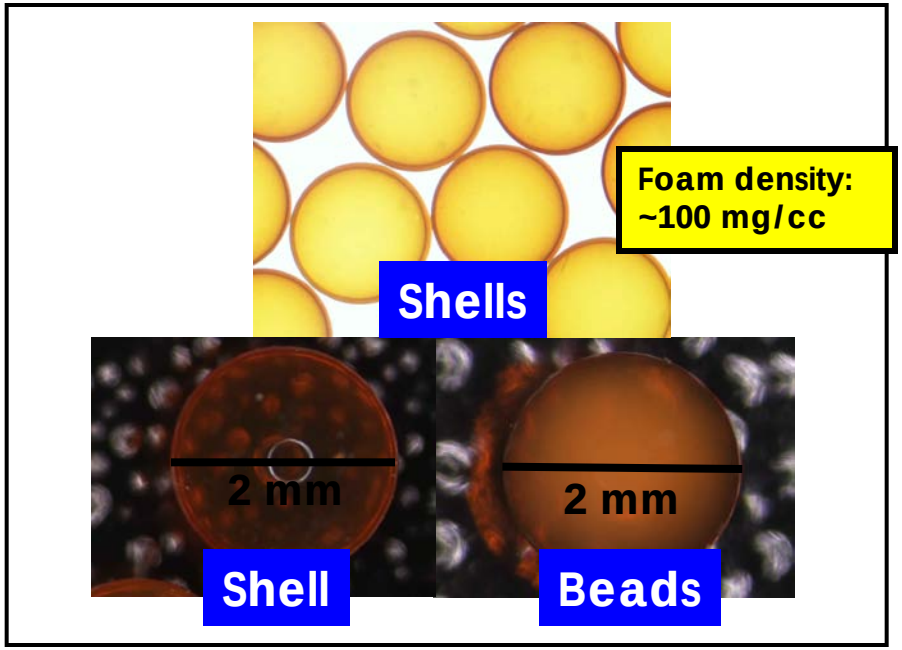
2. UV initiator replacing both Base & Acid catalyst (Mod 2)

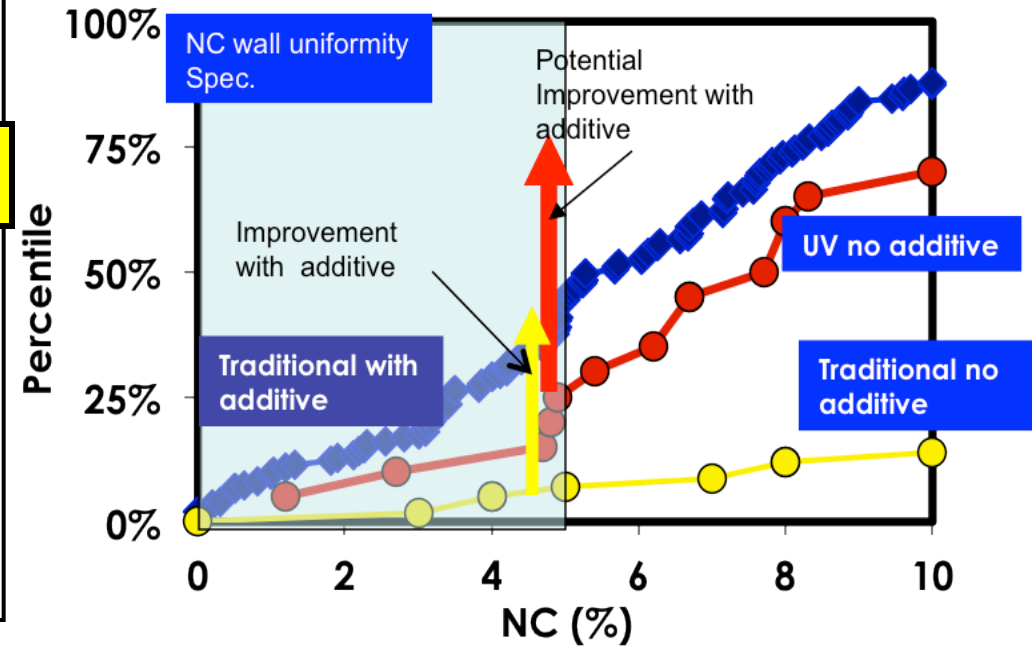
- Forms the primary particles & connects them to form the aerogel
- Conductivity ok for DEP

Free Radical formation using Irgacure 819 (BAPO)



Hollow R/F Shells were fabricated using a DG with UV initiated R/F using Modification 1A



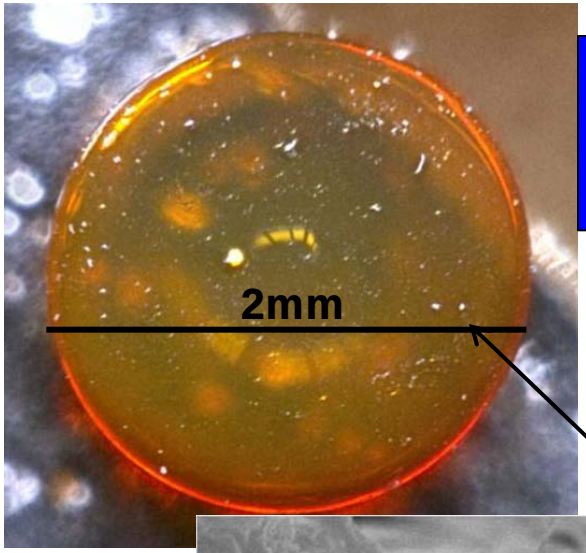


- **Gels in 15 min at room temperature**
  - No skin observed in shells & beads
- **Wall Uniformity (NC) is better than standard R/F (No Additives in oil)**
  - No temperature/density issues (70 ^{0}C)
  - *Additives can improve wall uniformity yield

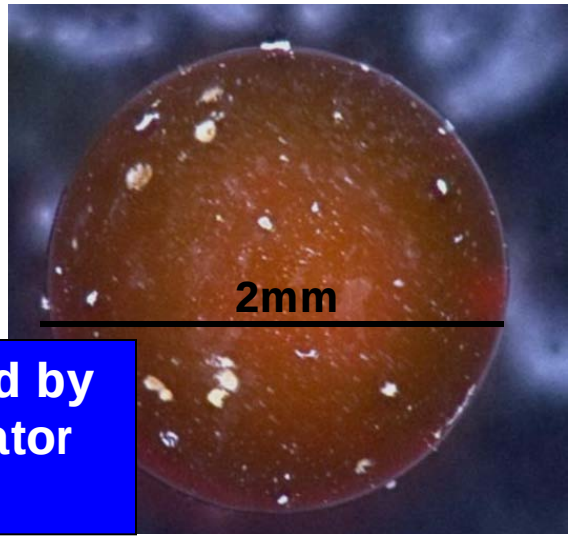
Potential to improve wall uniformity with addition of polymer additives

* R.R. Paguio, *et al.*, *Fusion Sci. Technol.* 59, 1, 199, (2011).

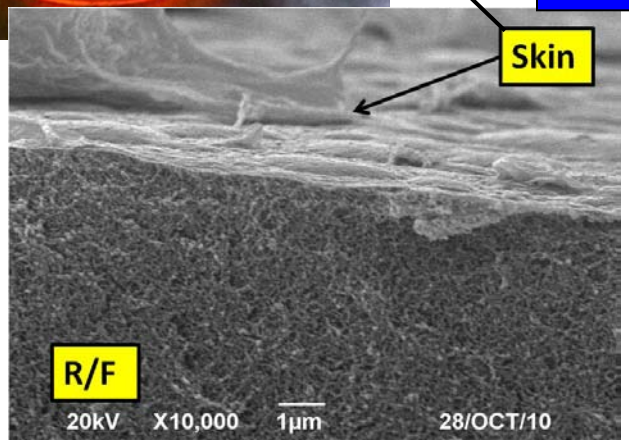
Hollow R/F Shells can be fabricated using a DG with UV PAG (Modification 1B)



R/F shell from the droplet generator method



R/F bead fabricated by the droplet generator method

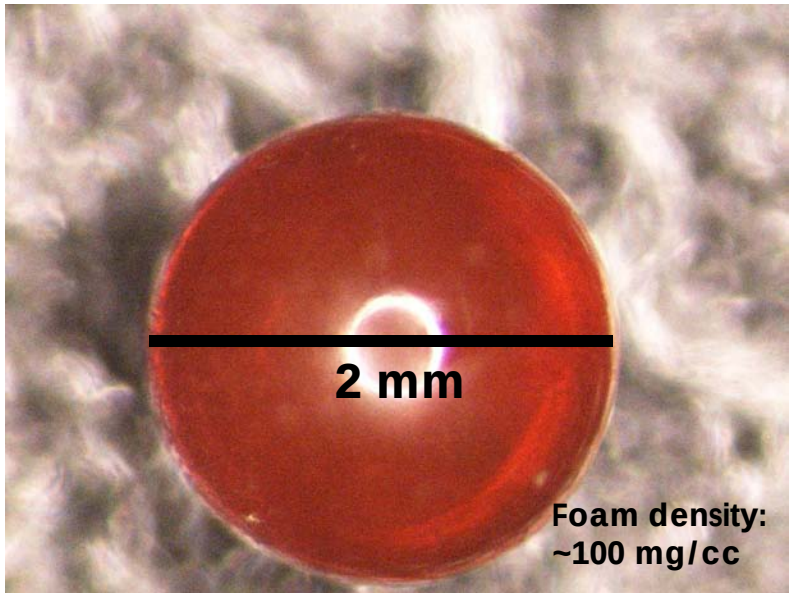


- **Gels in 15 minutes**
- **Skin on the R/F shells appear less than R/F beads**
- **Possible skin formation causes**
  - How sol was exposed (More even exposure)
  - Concentration of the PAG

GENERAL ATOMICS

Fabrication of solid R/F beads using DG using UV initiated R/F using Modification 2 (Replacing Acid & Base)

- **Gels in 20 min in DG**
- **Survives gelation process but the Shells do not survive DG process**
  - Unstable emulsion (Beads) in the curing process due to tumbling
  - No skin unlike the single bead test
- **This Sol is compatible with DEP**
  - Low Conductivity ($3X10^{-3}$ S/m)



<u>Can still work for DEP</u>

No Rotation: Stable Emulsion which gives emulsion a better chance to survive the curing process

Successfully fabricated hollow R/F aerogel shells with the traditional DG using free radical UV photoinitiators & PAG

- **Droplet Generator Shells using (Mod 1A) has most promise**
  - No Skin, 25% yield meet wall uniformity
  - Additives should help increase wall uniformity
  - Increase production throughput
    - Cures faster then traditional R/F
- **Mod 2: Sol survives gelation process to form beads**
  - Emulsion not stable to survive DG process
  - DEP emulsion more stable, increasing survivability VS DG
  - Create a more stable emulsion to survive in DG

<u>Future Work</u>: Create a centered R/F shell with UV R/F Modification 2 technique using the DEP process

DEP Presentation by G. Randall; et al., Wed. PM-1:25PM