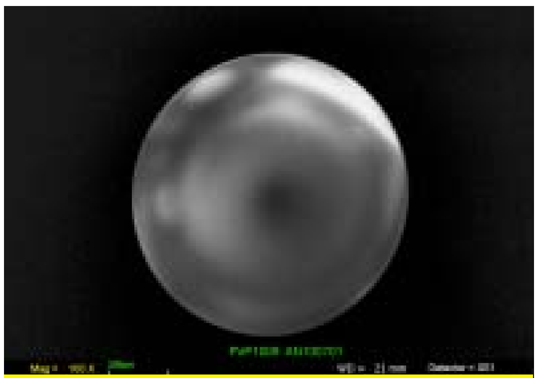
Foam Shell Fabrication R&D at General Atomics



PVP Coated RF Shell

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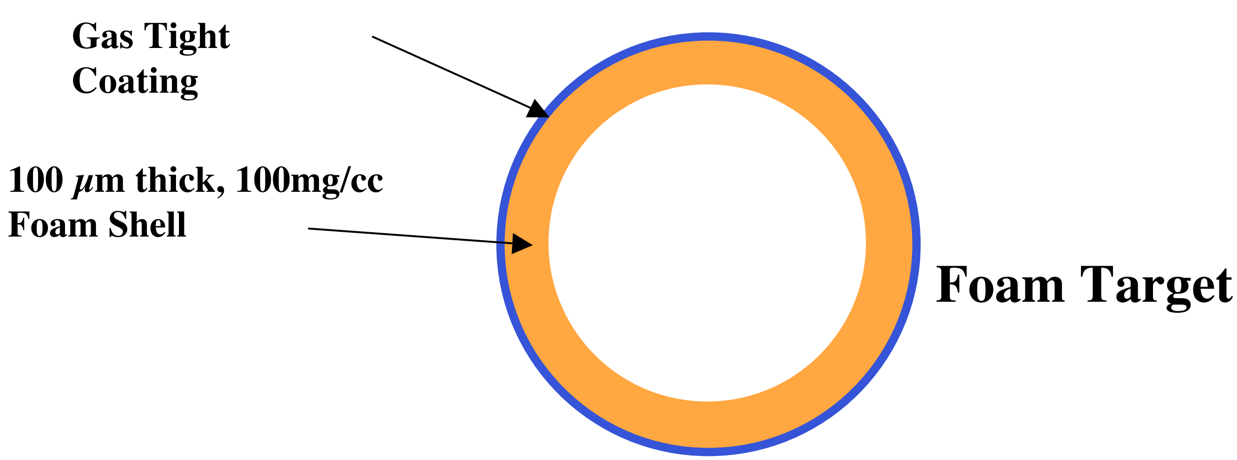
GDP Coated RF Shells

Presented at the 2nd US-Japan Workshop
2-3 February, 2003
General Atomics, San Diego, CA

Goal of Work:

Fabricate foam shells for U of R LLE- direct drive

≈ 100-250 mg/cc
≈ 1 mm diameter
≈ 30-100 μm wall
Permeation seal barrier- ≈ <1 - 3 μm
Implosion insensitive to as much as 30 at. % oxygen



Foam shell fabrication history when our work began:

- Takagi, et al (ILE) - methacrylate-based chemistry TMPT, EGDM- early '90s- *W/O/W system*

- LLNL/Schafer- reproduced ILE results- mid 90's

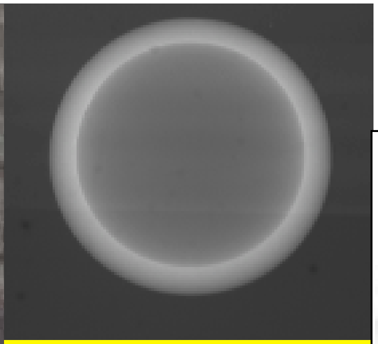
- LLNL/Schafer/ Soane -Resorcinol-Formaldehyde (R/F) reverse oil/water/oil system, late 90's- *"reverse" O/W/O system*

- Schafer- work had begun on DVB shell fabrication - *W/O/W system*

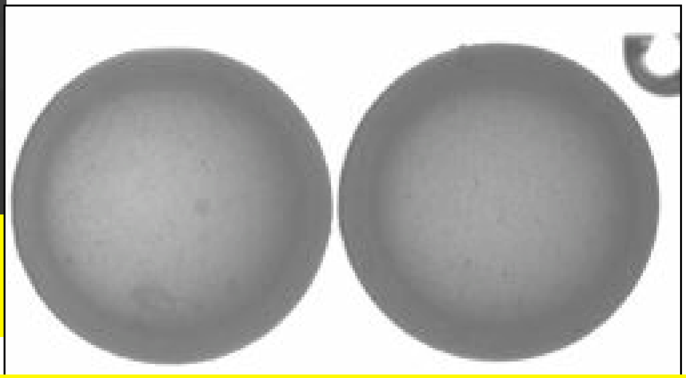
W/O/W systems were investigated initially

TMPT, EGDM, and DVB shells were made
These types of foam shells were opaque optically
Characterization is a big issue
R/F was chosen due to its transparency- reverse O/W/O system

DVB shells made using microencapsulation (~ 2 mm in diameter)



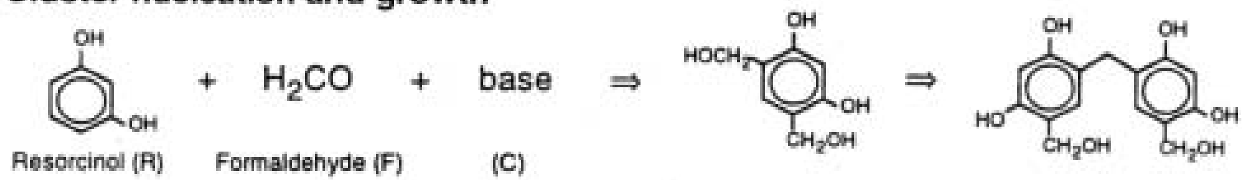
X-radiograph of a DVB shell



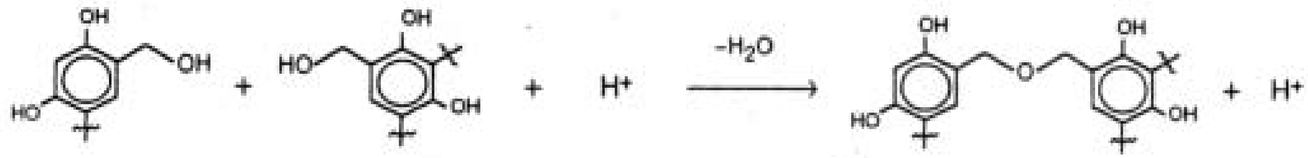
DVB shells are transparent only in liquid

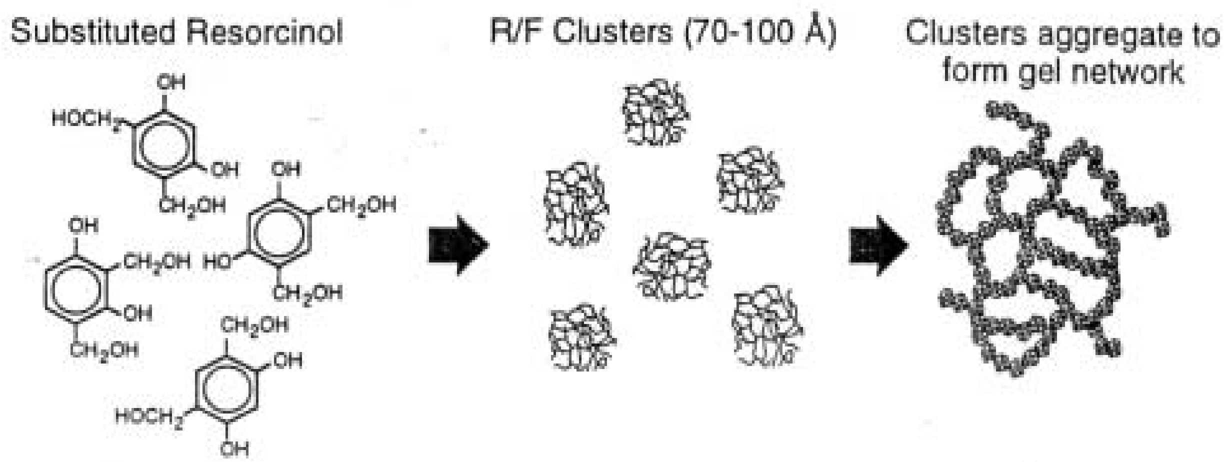
Resorcinol-Formaldehyde Chemistry Used

- Cluster nucleation and growth



- F/R controls functionality of substituted resorcinol
- R/C controls cluster size
- Cluster aggregation catalyzed by acid

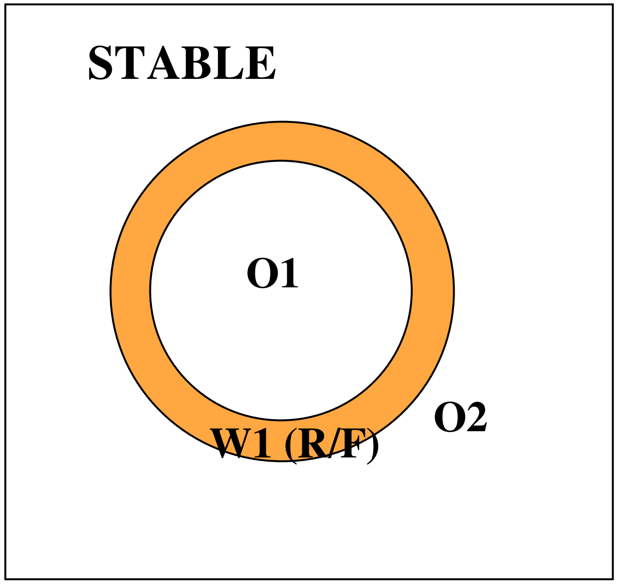


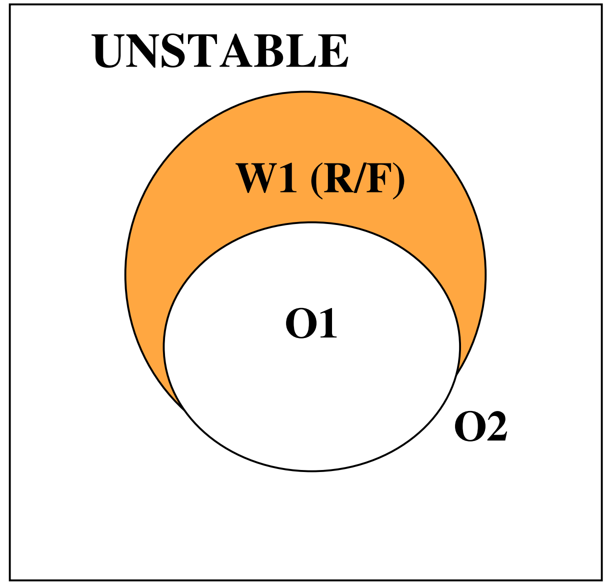


Challenges in R/F shell fabrication:

- Reverse system (O/W/O) - lots of oil!
- Stability of emulsion- LLNL experience
- Curing of shells without buckling
- Dehydration from curing shell by surfactant/agitation
- Shell buckling due to removal of inner oil core from shell
- Circular voids present in shell wall
- CO_2 critical point drying of shells
- Fabrication of spherical and concentric shells

LLNL work indicated problem with emulsion stability





LLNL experience indicated rapid gelation is necessary

Rapid gelation : not enough time for centering : poor NC

We found:

Proper density match leads to very stable preforms

Density matching is key for stable shell preform production

Density Oil-1 $\simeq$ Density W-1(R/F)

- Stable during droplet generation
- Stable during collection
- Stable during curing (can cure at room temp!)
- More concentric shells

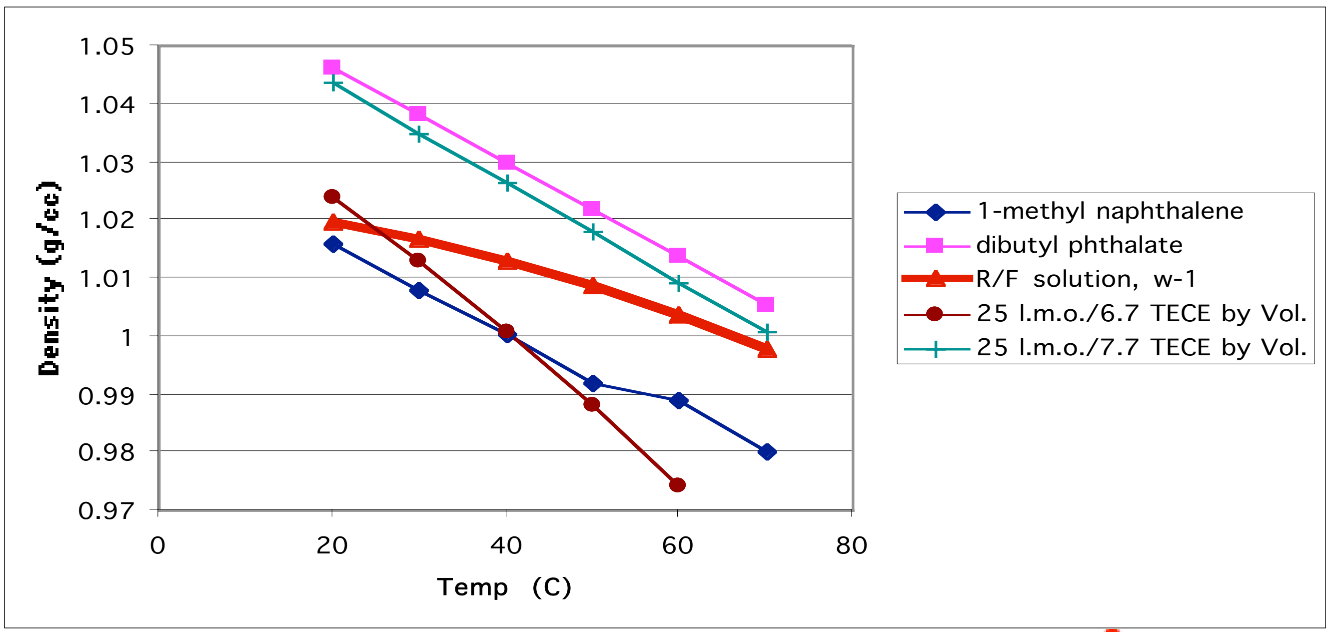
Density Oil-1+W $\geq$ Oil-2

- For proper agitation in rotavap flasks

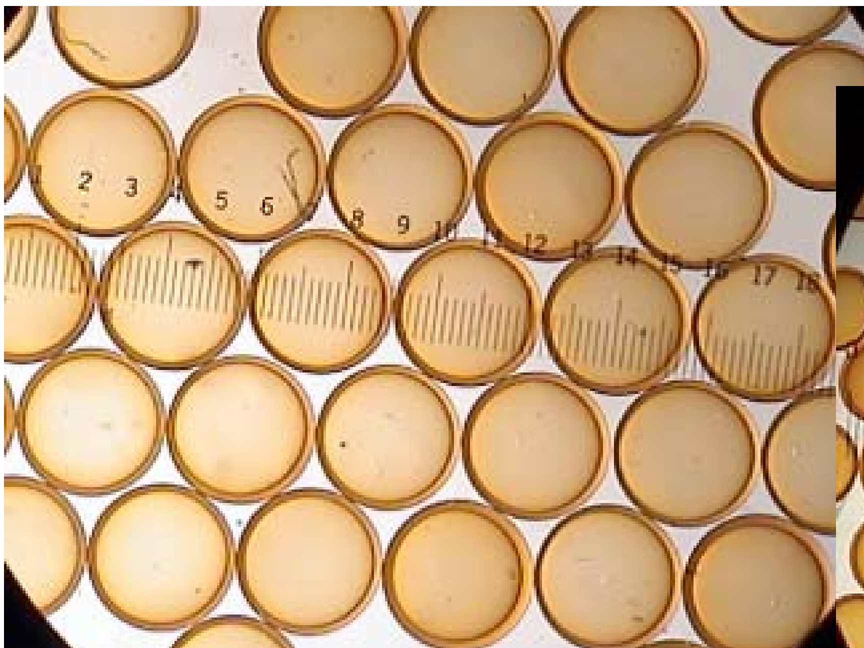
Candidate O1, O2 Phases

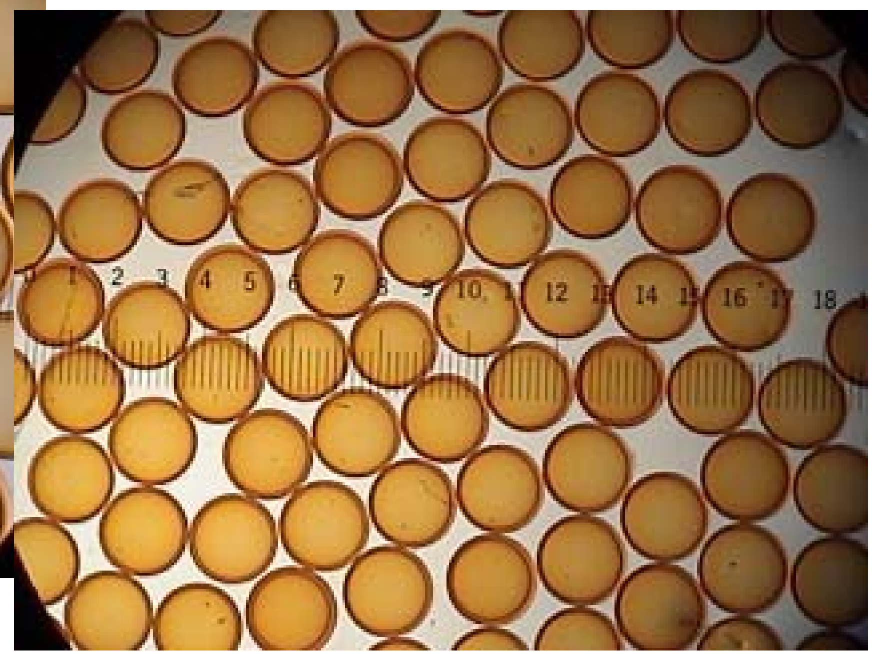
Liquid	Density (g/cc at R.T.)
R/F solution	**1.018**
Light mineral oil (l.m.o.)	**0.853**
Tetrachloroethylene (TECE)	**1.624**
Carbon Tetrachloride	**1.593**
DiButyl Phthalate	**1.042**
1-Methyl Naphthalene	**1.015**

As long as preform is stable at R.T. **: best to density match near curing temp.**



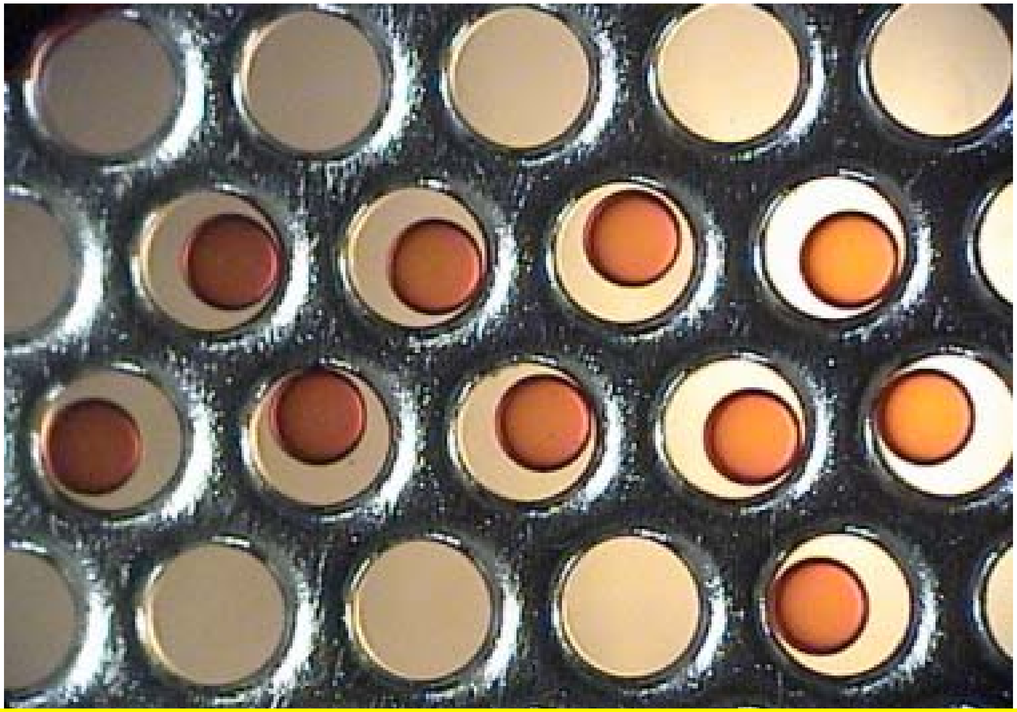
Batch views from R/F shell fabrication





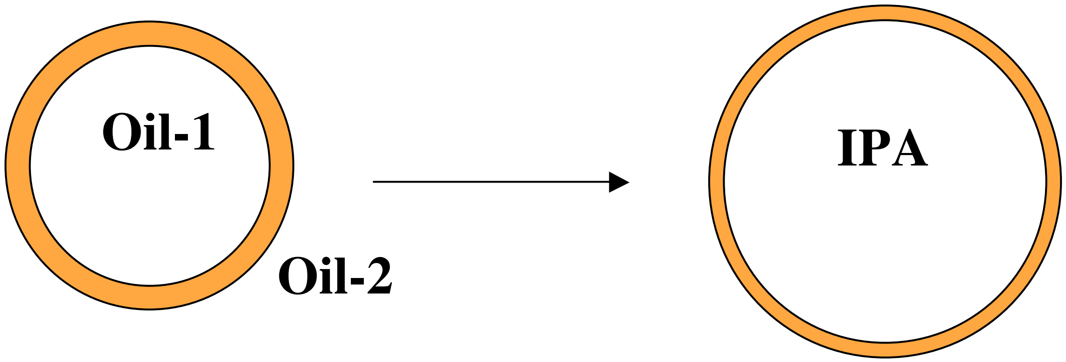
Shells are ~ 1000 μm diameter ≈ 80 μm wall thickness

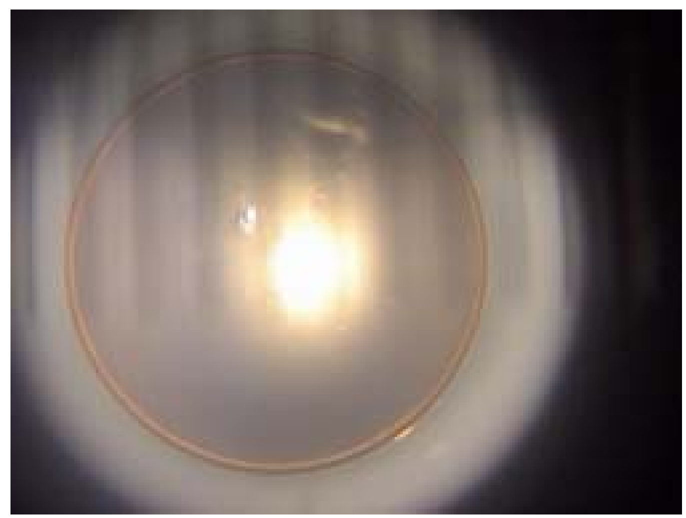
4π non-concentricity ≈ 4-8 %



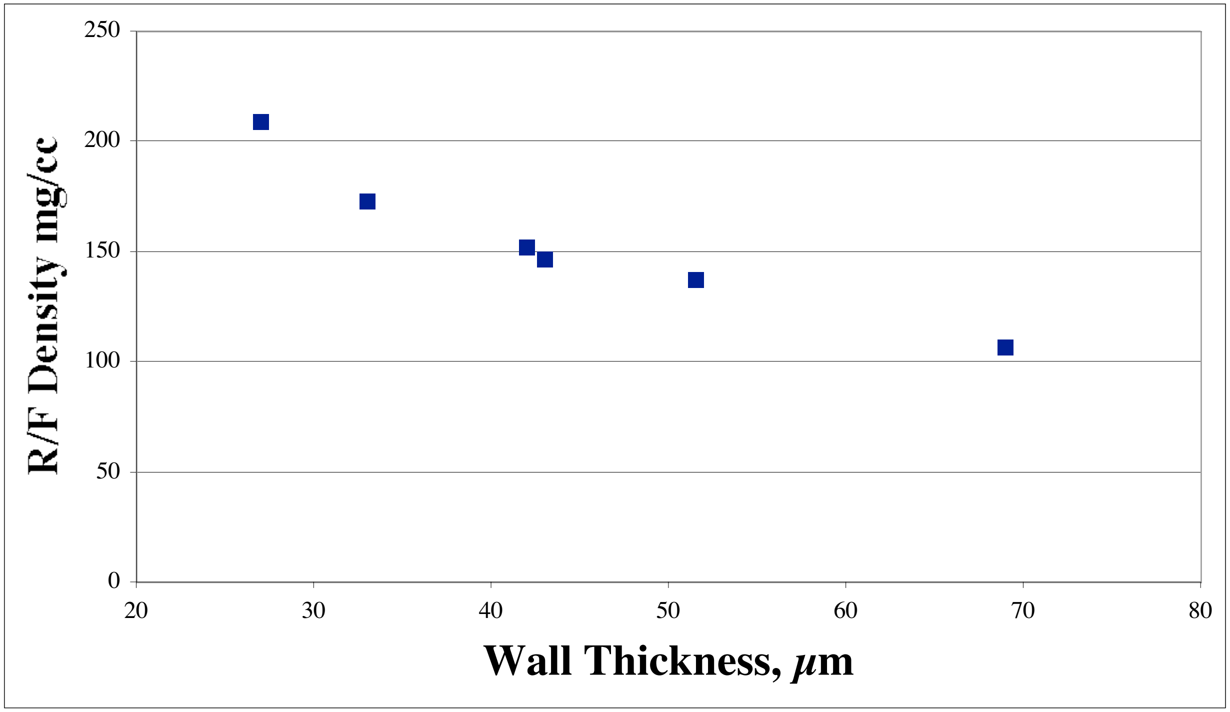
Optical view of dry R/F foam shells
(≈ 1200 μm OD ≈ 80 μm wall, 90 mg/cc)

Shell wall thinning in IPA can take place along with diameter expansion from insufficient curing





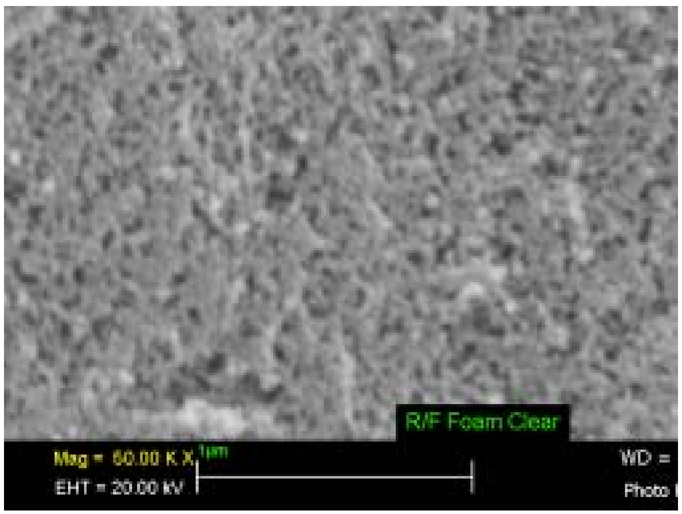
Density Increases as Wall Thickness Decreases for Shells within a Batch



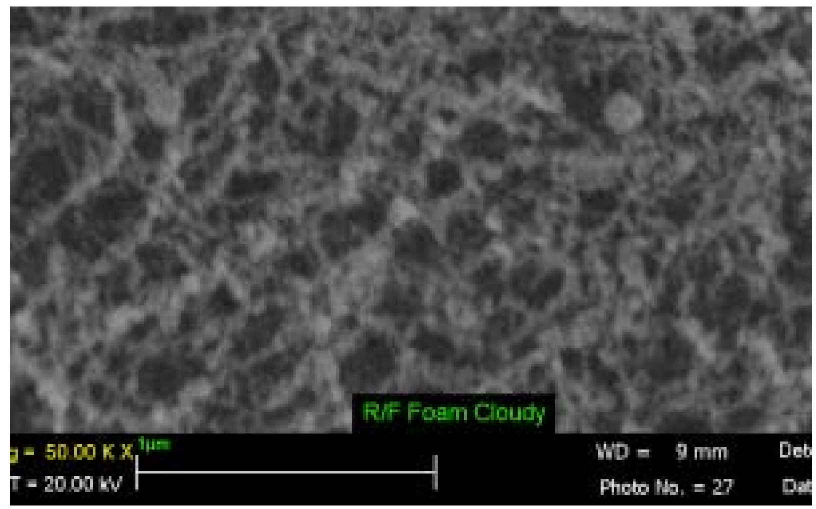
Extended curing is critical

GENERAL ATOMICS
AND AFFILIATED COMPANIES

Catalyst Concentration Observed to Influence Cell Size and Opacity of RF Product



low benzoic acid concentration -> clear foam



high benzoic acid concentration -> cloudy foam

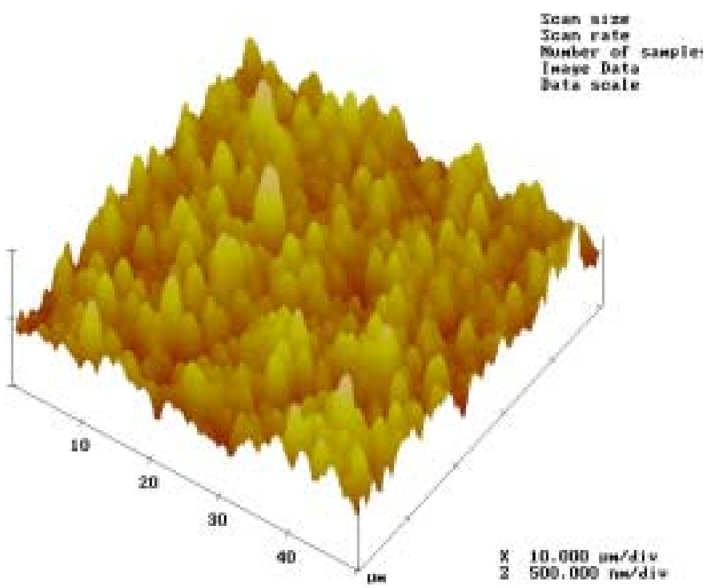
Progress Made in Overcoating of RF Shells

- **GDP coating of dried RF shells, 2-8 μm layers**
  - coating retains Argon-permeation similar to GDP
  - GDP/RF shell has higher buckle strength than GDP alone

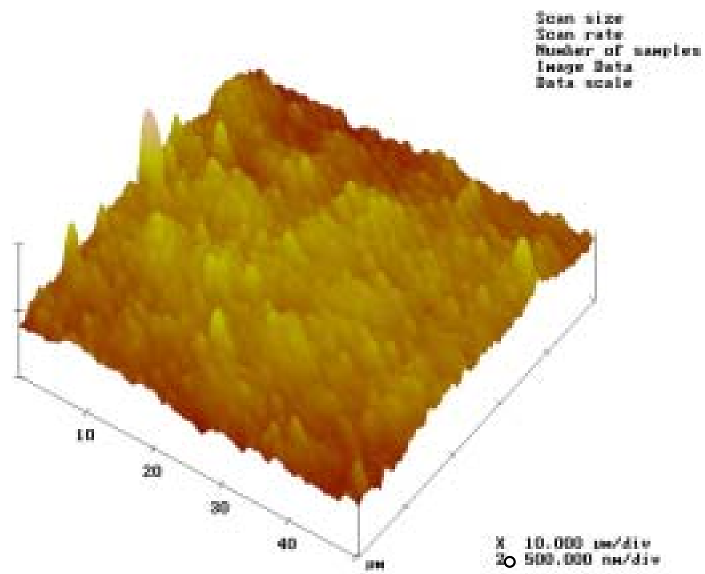
- **PVP coating of RF shells, 1-4 μm layers**
  - coating retains Argon- permeation similar to GDP
  - PVP coated RF shells have extremely high strength

GDP coating was initially used as an overcoating for R/F

Relatively smooth coatings have been deposited on R/F shells



RMS = 810 Å
75 mtorr

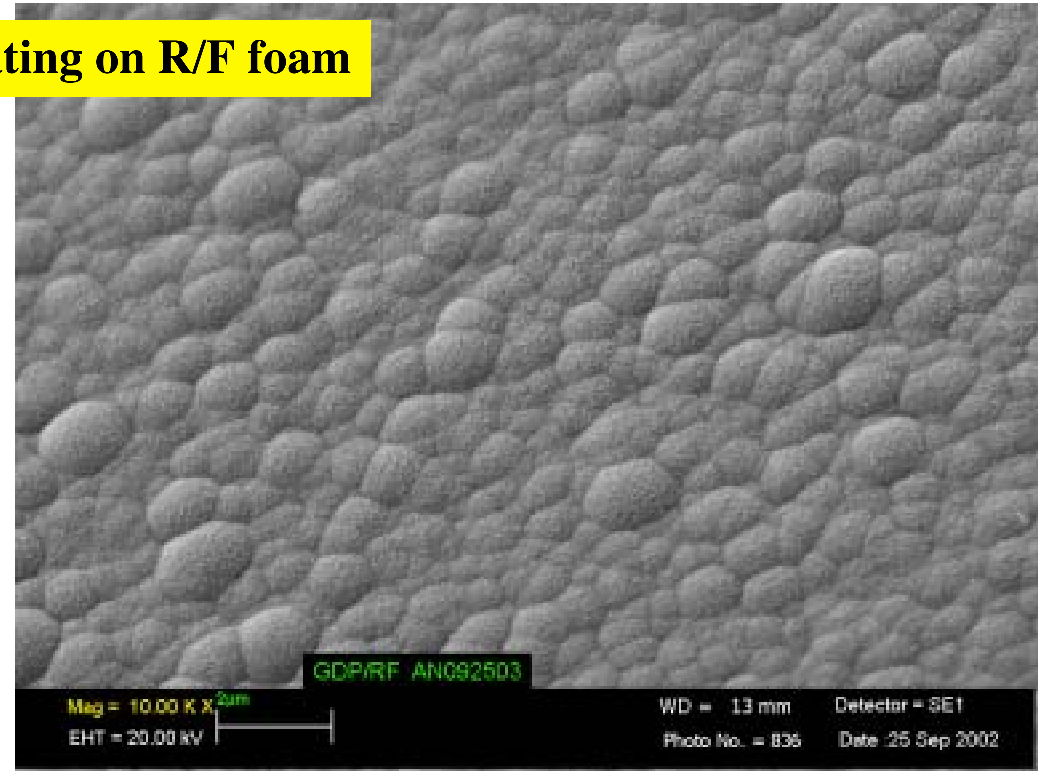


RMS = 410 Å
500 mtorr

Higher pressure coatings lead to smoother surfaces- better pore closure

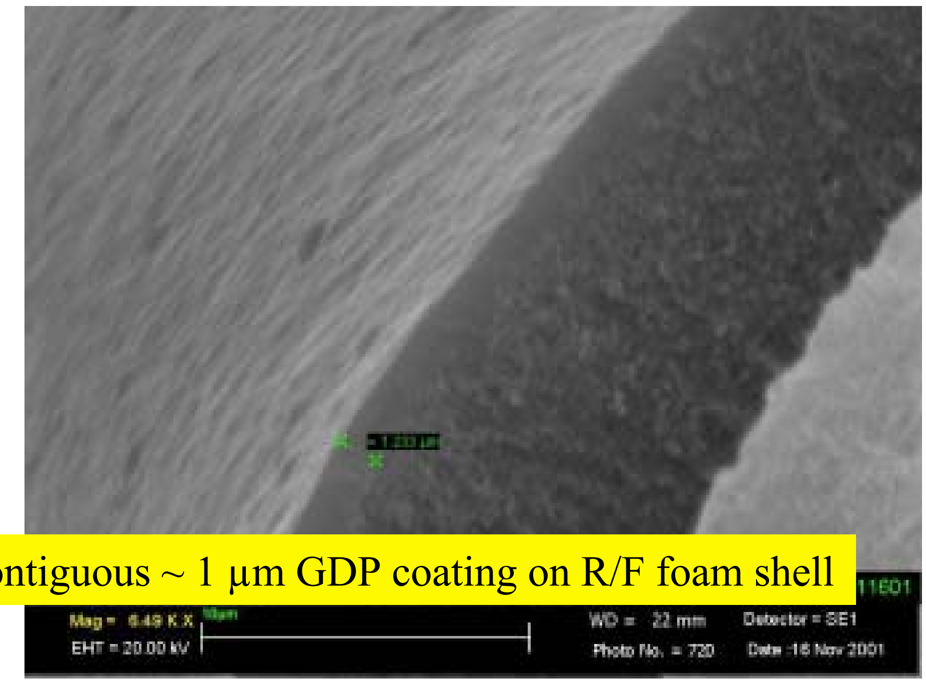
GDP Coating of R/F shells

≈ 4 μm GDP coating on R/F foam



• GDP does close up R/F foam pores even at 2-3 um thickness

GDP coatings do act as permeation seal barrier



contiguous ~ 1 µm GDP coating on R/F foam shell

Ar Gas Retention Testing by XRF

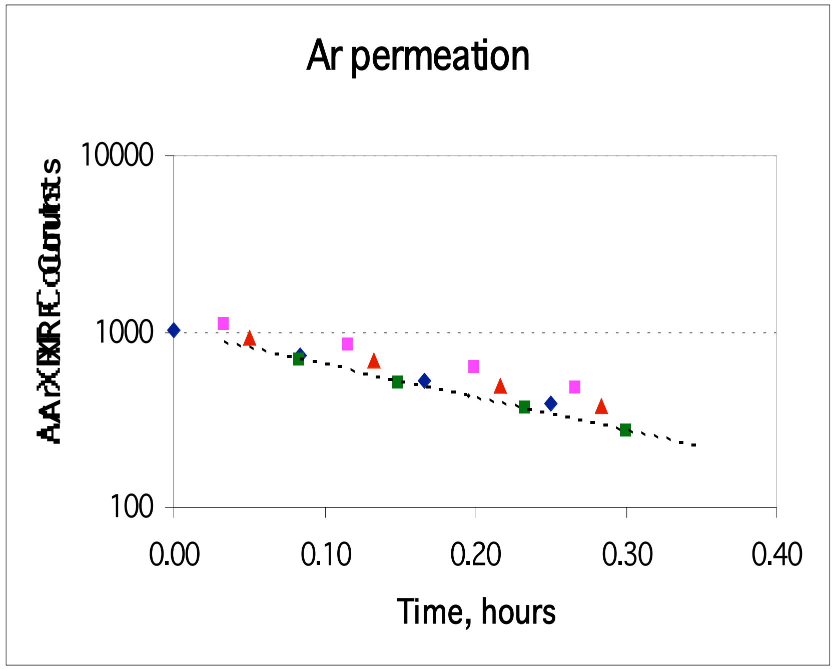
GDP thickness 3.3-4.7μm

- 49 GDP coated shells tested
- 34 shells held Ar by XRF

~70% shells tested had gas-tight GDP coating

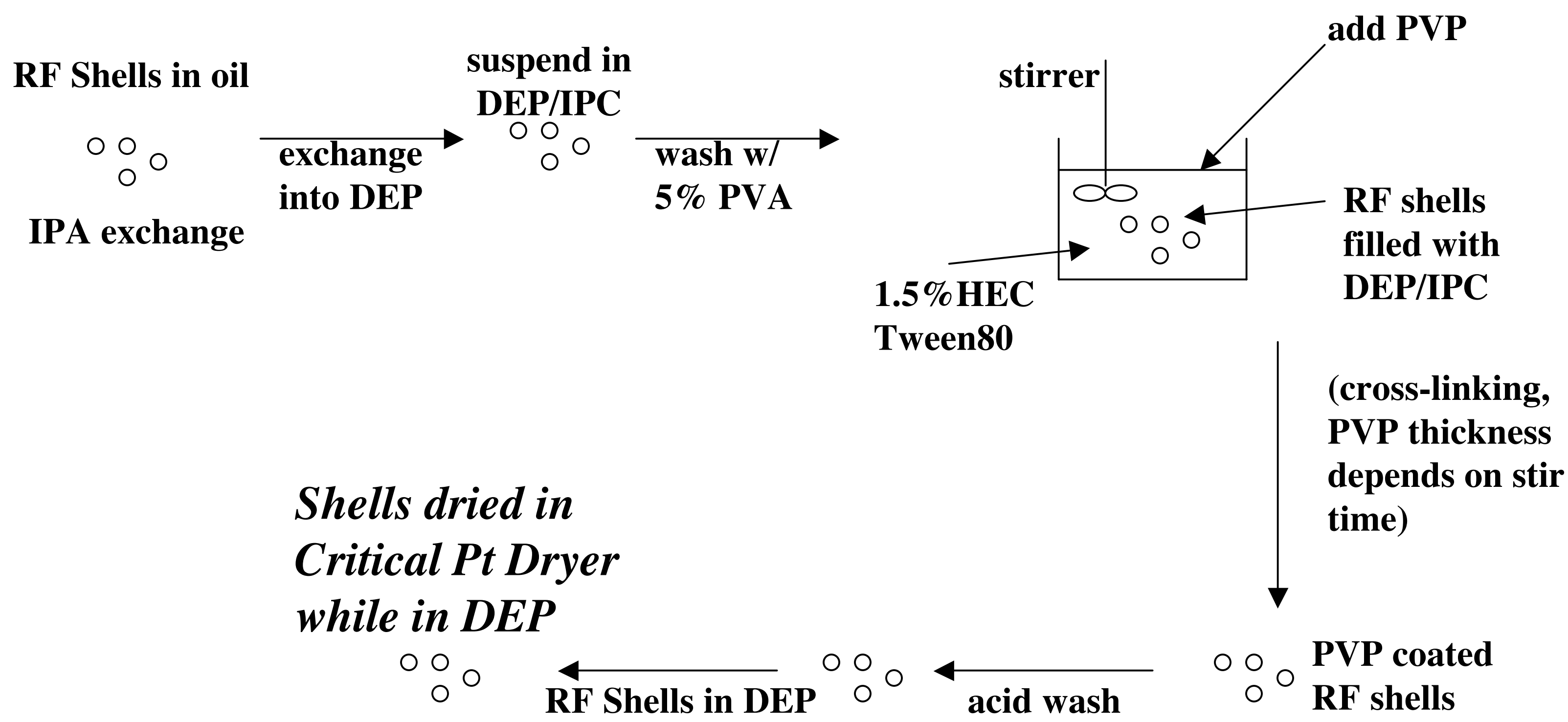
compare:
PVP-coated 90%

GDP coatings on R/F shells provide a gas barrier



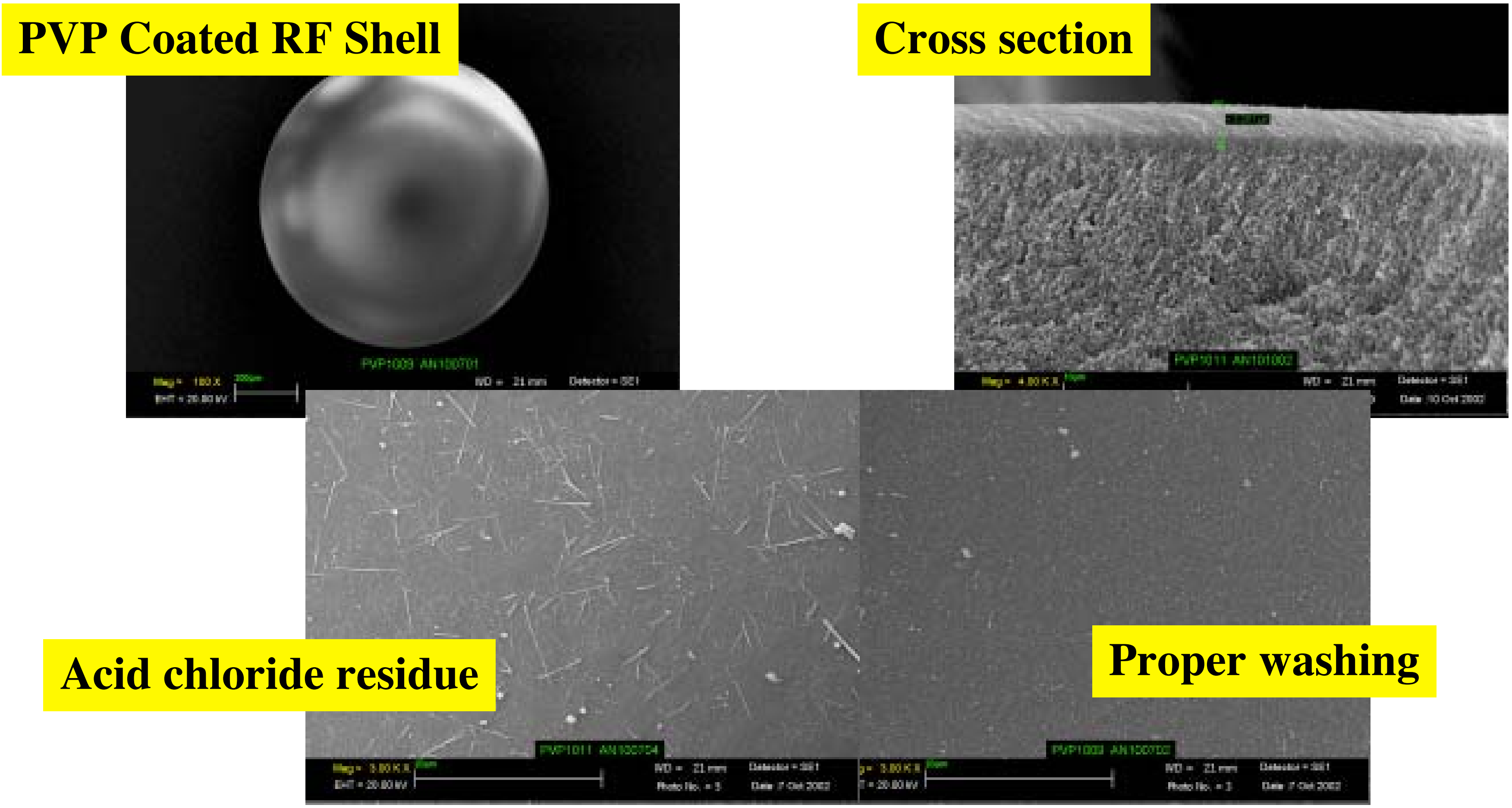
Argon permeation data provided direct evidence of gas retention of GDP coated shells

Polyvinylphenol (PVP) Coating Process



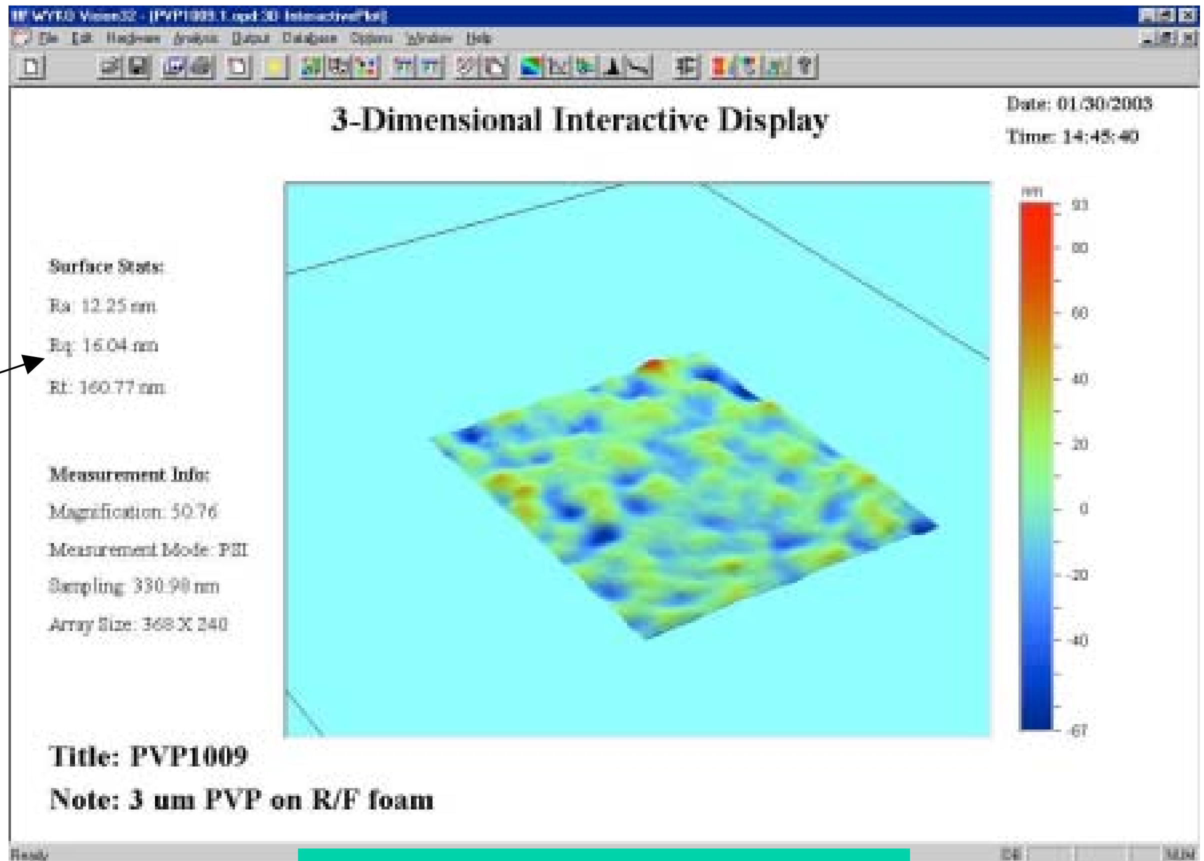
IPA isopropyl alcohol
DEP diethylphthalate
IPC isophthaloyl dichloride
HEC hydroxyethylether cellulose

SEM examination of PVP Coated R/F Shells



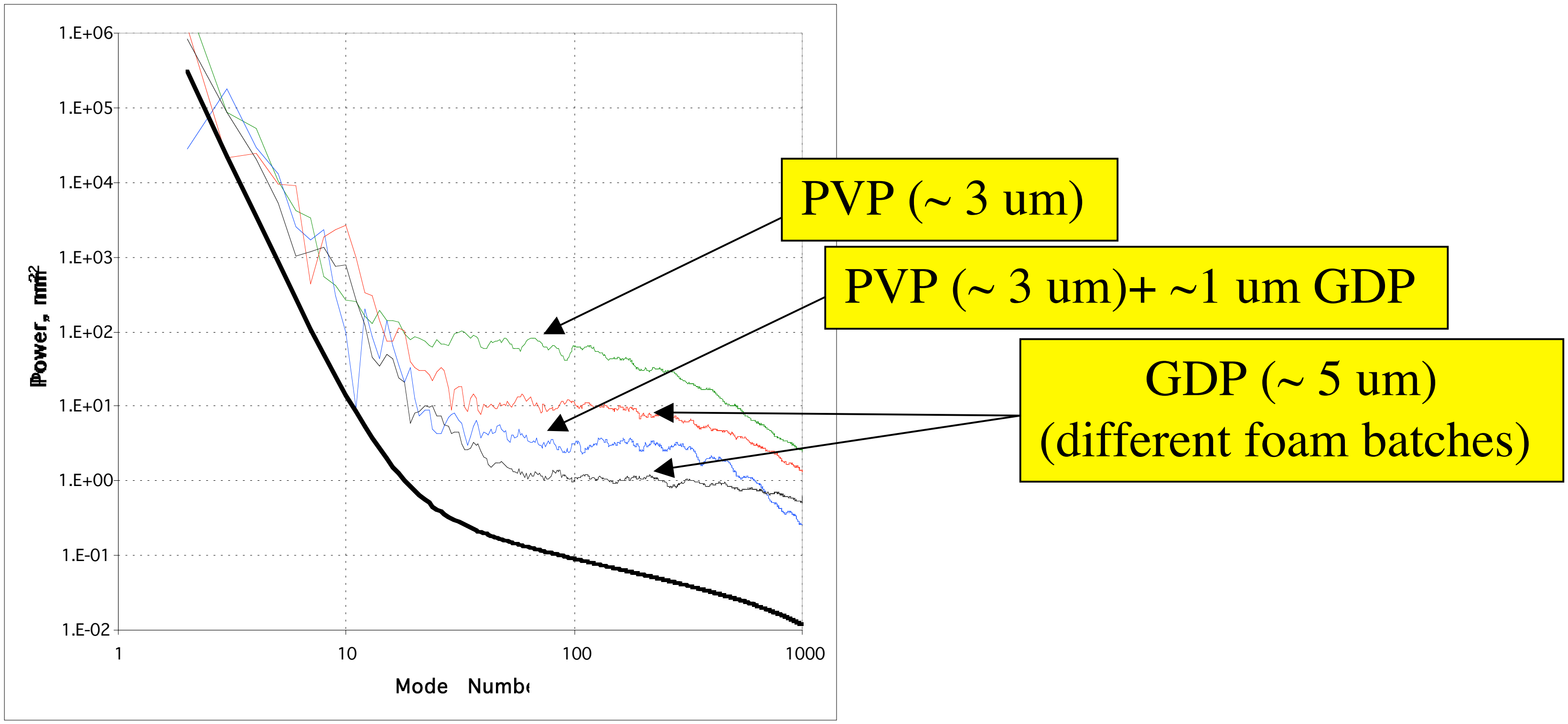
Interferometric surface roughness measurement

RMS= 16 nm



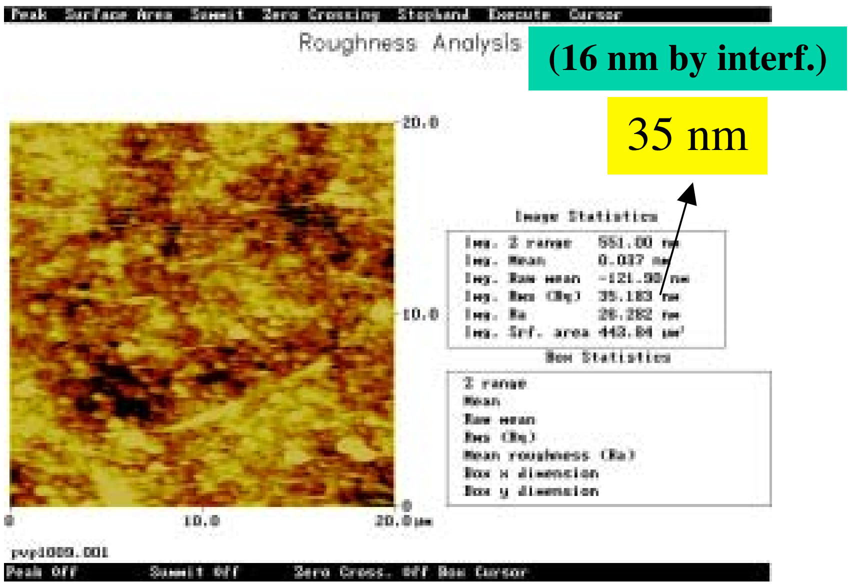
WYKO interferometer data

PVP coatings also have high mode roughness comparable to GDP coatings

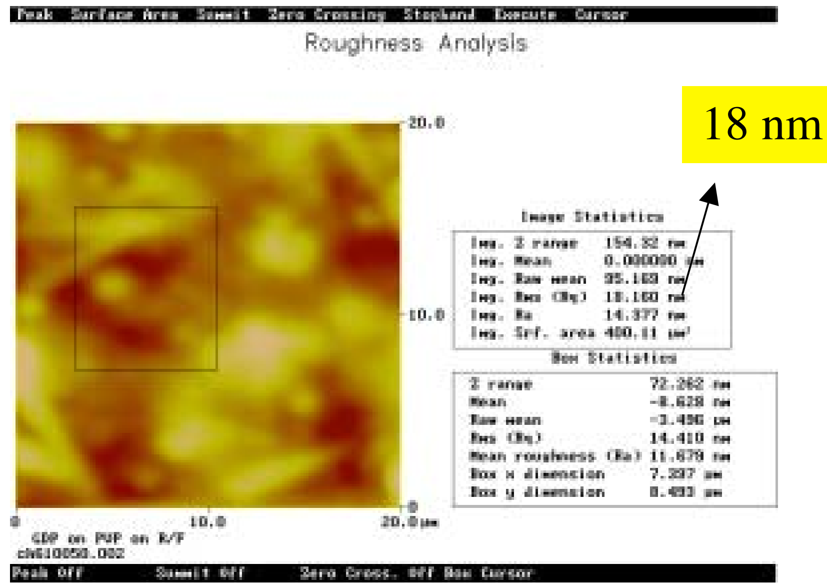


- PVP coatings have rougher high mode power spectra compared to GDP coatings!
- AFM patch scans, however, indicate somewhat better PVP background (high mode) surface finish
- GDP overcoat of PVP smoothes coating as seen by spheremapper and patch AFM

PVP coated shells were smoothed by GDP overcoating



PVP Coated R/F shell



GDP coated PVP Coated R/F shell

PVP Coated RF Shells are observed to have higher buckle strength

Shell	Norm Buckle Strength (psi)
Std-GDP	1.5
Std-GDP coated RF	2.0
PVP coated RF	*3.0*

Normalized buckle strength corresponds to 900μm OD shell with a 1μm Wall

Std-GDP is standard Glow Discharge Polymer, deposited at 75 mtorr

SUMMARY

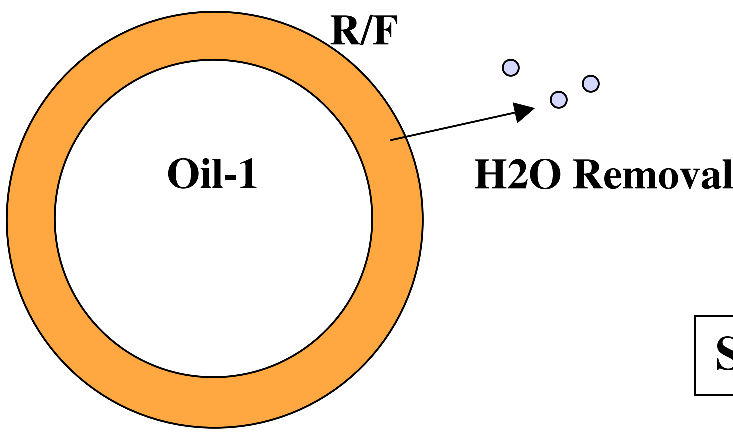
• Significant Progress on RF Shell Fabrication and Coating

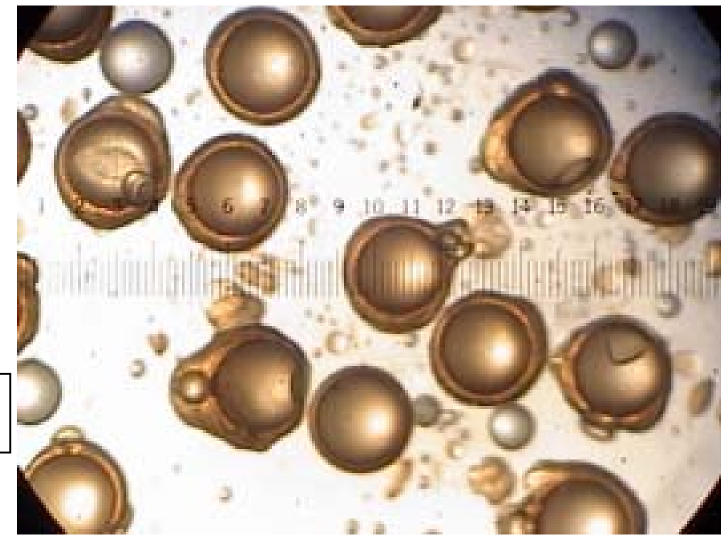
• Shells of ~1.0 mm diameter, 60-100 μm wall, and ~100 mg/cc density have been fabricated and coated by GDP and PVP

• Several overcoated shells were tested for gas retention, and sent to LLE - One shell (5 um GDP overcoat) was filled with 1000 atm D2 and cooled to cryogenic temperatures - Shell survived

• D2 was frozen and layered - first time in U.S. using foam shell

During curing dehydration of shells can occur

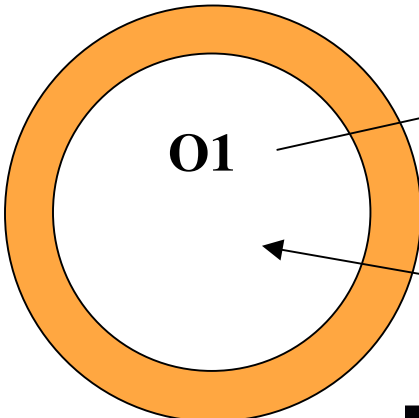



Shells in Oil-2

Dehydration can be caused by;
- Too much surfactant in O2
- Vigorous agitation during curing
- Too long of cure
- *Happens in mineral oil- (low water solubility)*
- Wrinkling is somewhat smoothed out in IPA

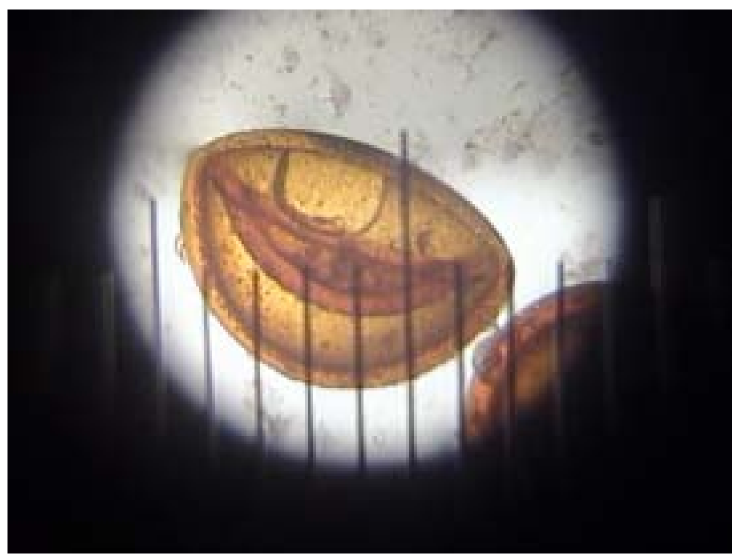

Shells in IPA

Rapid transport of O1 phase from interior during curing or IPA exchange can buckle shells



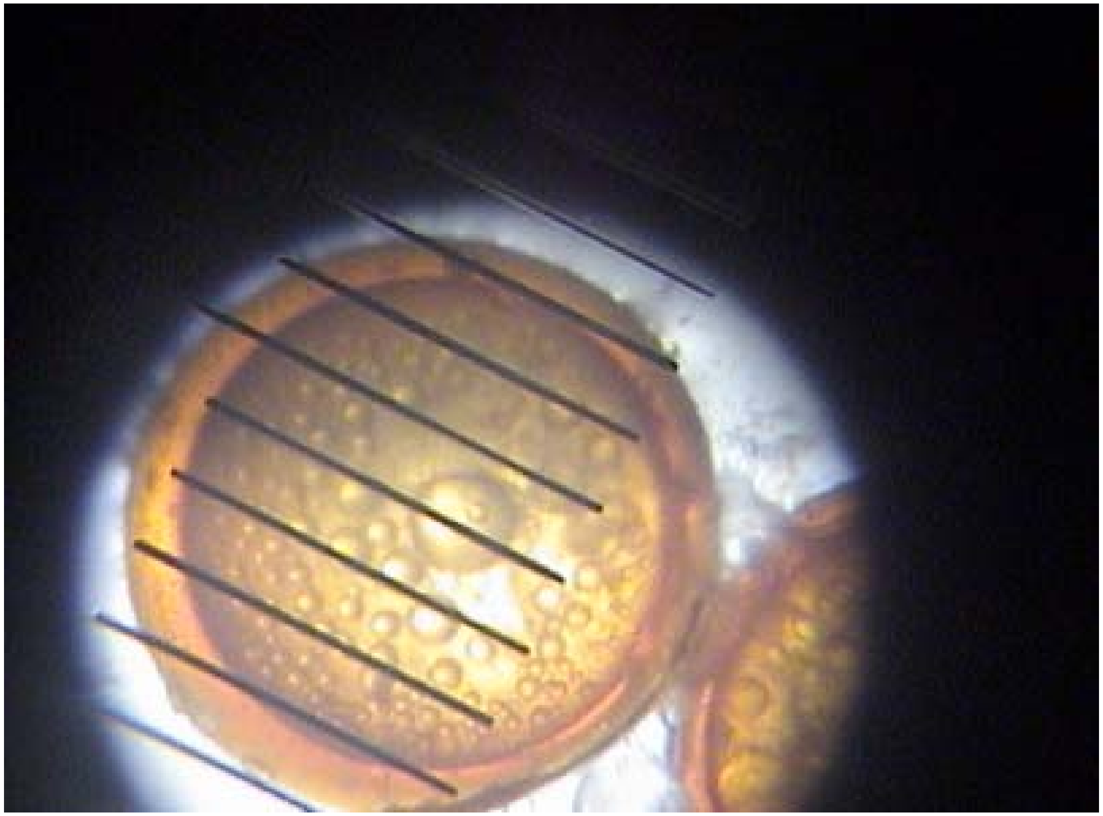
O1: dibuytl phthalate (fast)
1-methyl naphthalene (fast)
light mineral oil & mixtures (slow)

O2 or IPA (Isopropyl Alcohol)

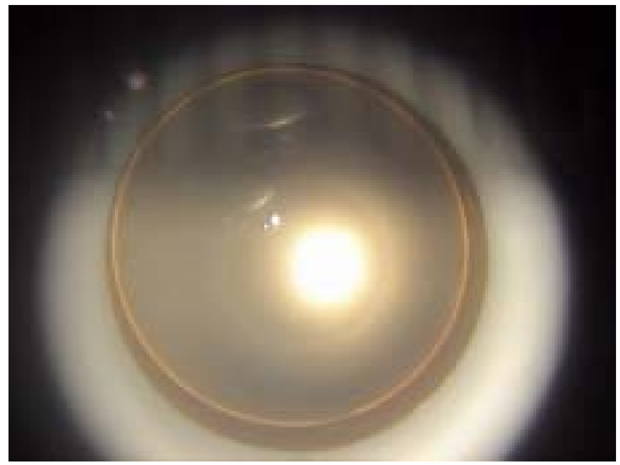


1-methyl naphthalene used as O1

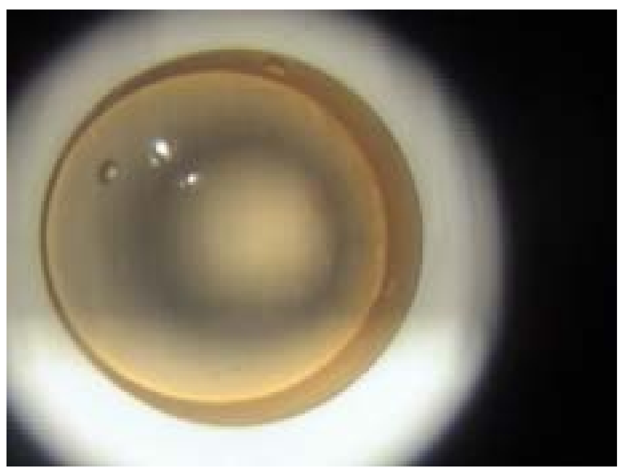
Slow removal of O1 core from cured shell in IPA

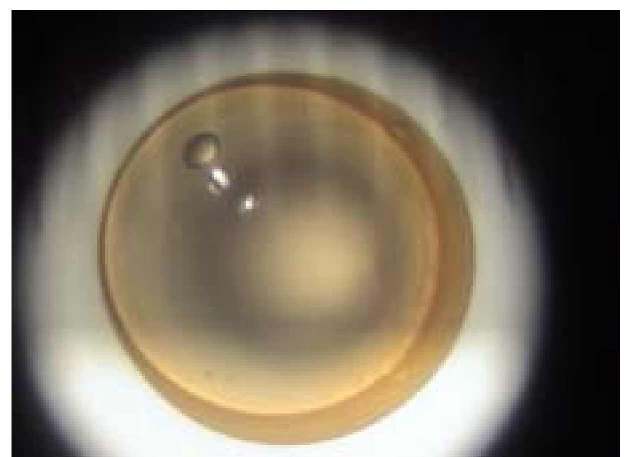


We have observed the growth of circular voids in shell wall upon exposure to IPA



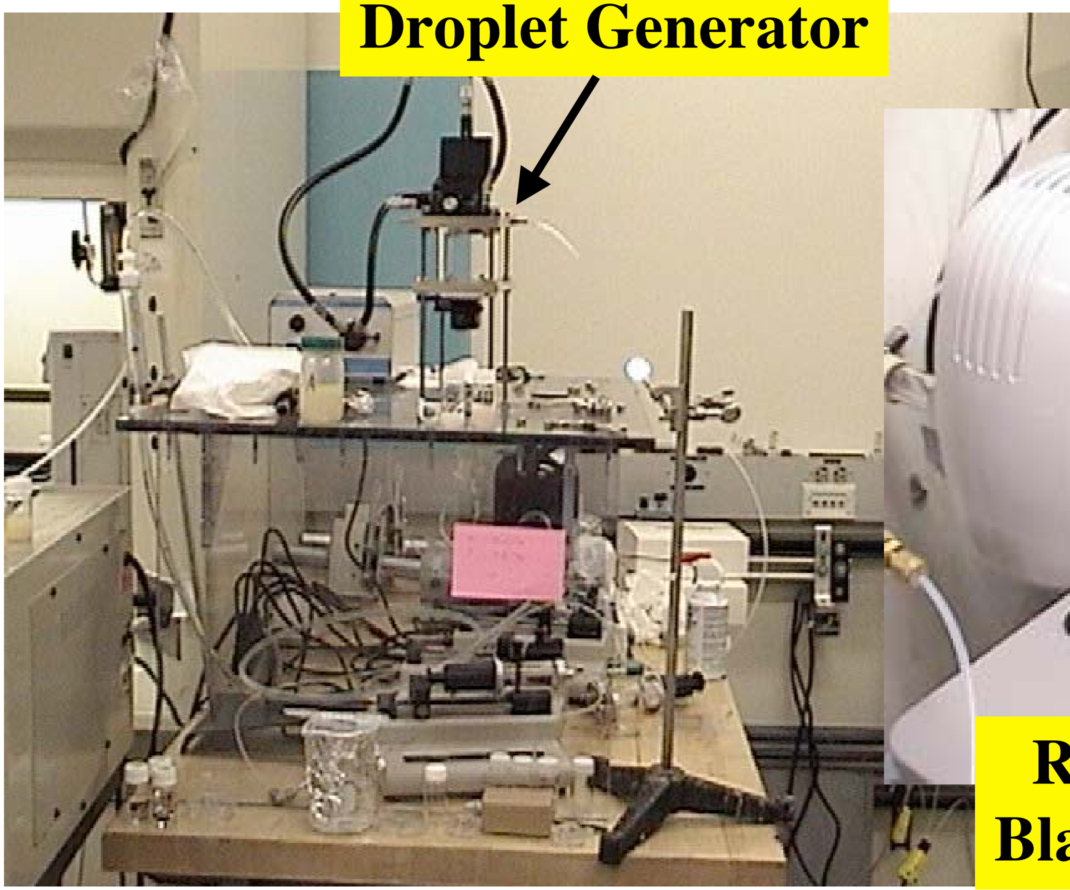
Side-Views of Circular Voids in Shell Wall

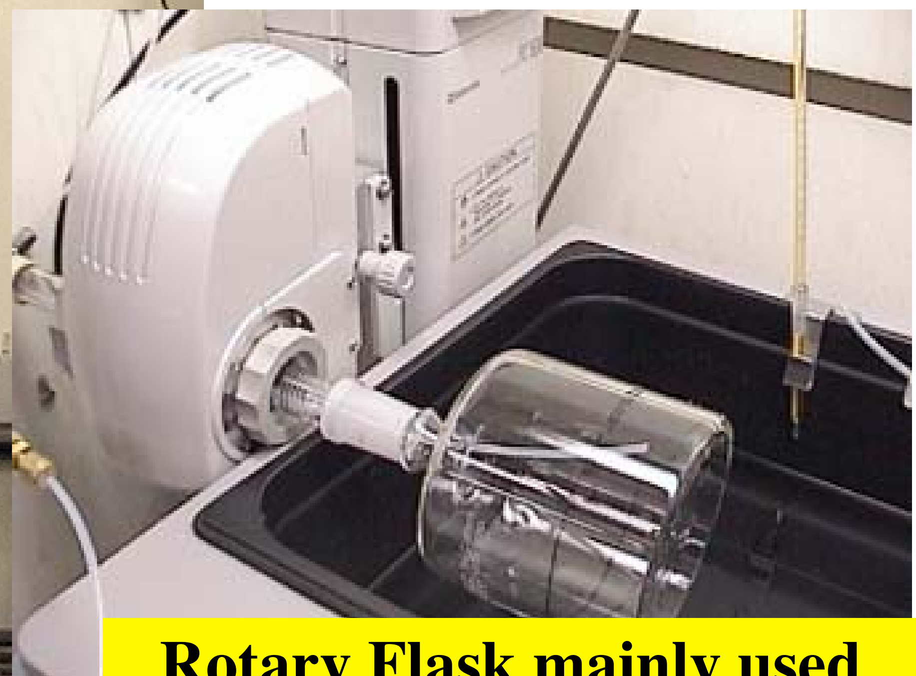




Droplet Generator used for R/F Shell R&D
Triple Orifice Osaka Design

Droplet Generator





Rotary Flask mainly used
Blades System also examined