Advances in Si-GDP, Ge-GDP and Glass Capsule Fabrication

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22nd Target Fabrication Meeting
Las Vegas, Nevada
March 12-16, 2017

This work performed under the auspices of the U.S. Department of Energy by General Atomics under Contract DE-NA0001808 and by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 and General Atomics IR&D Funds
Recent development efforts to be discussed

1) Partially densified glass capsules from silicon doped glow discharge polymer (Si-GDP) for use in Pushered Single Shell (PSS) experiments

2) Fully deuterated germanium doped (Ge-GDP) capsules - also for PSS experiments

3) Improved route to manufacture glass capsule fill tube assemblies (CFTA’s) using the Si-GDP -> glass process

4) Method for manufacturing machined “keyhole” GDP/Si-GDP dual layer capsules for shock-timing experiments

5) Latest progress in expanding the range of glass capsules that can be made via the Si-GDP process
Plan was to start from the Si-GDP to Glass Conversion process

Phase I: Using normal dopants (TMS and T2B) show process will work
Phase II: Replace TMS with DTMS and T2B with DT2B in GDP process

Phase 1 Results: @ 490°C
Final Density 1.80g/cc
Silicon Density 0.66g/cc
OD ~1200 μm; 11 μm wall

SIMS shows there was Hydrogen present but cannot say quantitatively how much

TMS = tetramethyl silane
T2B = trans 2-butene
Decision: IR shows Deuterium lost during glass conversion

IR Spectra

Pre-glass conversion

C-D

C-H

O-H

Wavenumber (cm⁻¹)

4000  3000  2000  1000

Potentially of interest??

O-H

Partial glass conversion

Wavenumber (cm⁻¹)

4000  3000  2000  1000

Si-GDP (no D) (TMS; T2B; H₂)
Si-GDP (with D) (TMS; DT2B; D₂)

 Partially converted Glass from Si-GDP (no D)
 Partially converted Glass from Si-GDP (with D)

Project stopped/put on hold until further notice
Deuterated Ge-doped GDP capsules fabricated for Single Pusher Experiments

Phase 1) Replaced precursor gases H₂ with D₂; T2B with DT2B but keep normal tetramethyl germane (TMG)
- Refine coating parameters using normal TMG first to minimize cost

IR Spectra:

Post PAMS pyro

C-D

1400 μm

C-H
Phase 2) Replaced final precursor gas Tetramethyl Germane (TMG) with deuterated TMG

IR Spectra:

**Fully Deuterated GeGDP (3.7μm) on PAMS mandrel (12.5μm)**

(PAMS background removed)

IR Spectra of Deuterated Ge-doped GDP

1. **C-H (PAMS)**
2. **C-D**
Quantitative Deuterium content can be measured by Nuclear Reaction Analysis (NRA) with 3He beams

3He NRA for deuterium profiling

D(3He,4He)H1

3He + D = 4He + 1H

Q = 18.4 MeV

Method calibrated using Deuterated polystyrene

Best NRA estimate indicates ~65% D enrichment for DGeGDP sample but error bar is still large

Problems to address in upcoming NRA experiments

• Beam-induced D/H loss
• Noisy signal
• Shells -> Geometrical effects
Initial CFTA capsule drilling trials with Drop-Tower (DT) glass capsules had limited success.

**Issues with DT Glass**

- Poor coupling of laser with Glass capsule
- Irregular shaped holes
- Micro-cracks common around hole

Micro-cracks can lead to failure at pressure.
Capsules starting from Si-GDP overcame the issues encountered with Drop-Tower glass

Laser hole drilled in Si-GDP mandrel prior to conversion to glass

- Laser coupling with Si-GDP much better than DT Glass
- Much more uniform and round hole shapes
- No micro-cracks around hole
Goal: Produce capsules consisting of GDP/Si-GDP that can be machined without cracking or deformation

4 Methods Considered:
1) PAMS pyro followed by machining
2) Machining followed by PAMS pyro
3) Machining follow by removal of PAMS using Toluene
4) Laser Drill small hole, short PAMS pyro, then machining
Substantial Issues with first three methods

1) PAMS pyro followed by machining
   - Hairline cracks in Si-GDP layer. No machining attempted. Likely due to shrinkage difference between Si-GDP and GDP during PAMS pyro

2) Machining followed by PAMS pyro
   - resulted in severely distorted capsule

3) Machining followed by PAMS removal using Toluene
   - Shells very fragile and difficult to process
Best route was laser drill small hole followed by short PAMS pyro and then machine.

Capsule after PAMS removal

Capsule after machining

Final Capsule after release from mount
First attempt to make the thick walled glass capsules (>35 µm) resulted in shards and hemispheres.

Hemi post PAMS pyro

Interface generated at Si-GDP coating breakpoint

Nearly intact inner glass capsule

450°C air pyro

Interface (above) and intact inner glass capsules (right) gave clues to changes to be made for next attempt.
No-breakpoint run went much better with mostly intact capsules even after conversion to glass.

Majority survived PAMS pyro intact.

Conversion in He/O₂ (530°C)

~1mm x 60μm SiGDP capsule

~700μm x 36μm glass capsule
Latest addition shows ranges of capsules made by the Si-GDP process continues to grow
These examples demonstrate we are dedicated to developing targets to meet the needs of the ICF community

1) Partially converted Glass Capsules doped with D
   – Possibly of future interest but more development needed

2) Fully Deuterated Ge-GDP
   – Successfully(?) made capsules >90% enrichment
     (experiment in progress to confirm)

3) Glass capsule fill-tube assemblies
   – CFTAs from Si-GDP glass solves issues associated with DT glass

4) Machined GDP/Si-GDP capsules for Shock-timing experiments
   – Successful technique developed to provide high quality mandrels

5) Thick-walled (>35μm) glass capsule for PSS experiments
   – Mostly successful and extends the range of glass capsules that can be made by the Si-GDP process