Proceedings

Compiled by: Larry R. Foreman
and Jean C. Stark

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The meeting of specialists in target fabrication for inertial confinement fusion was the tenth such meeting in the spirit of a high level workshop on target technology. Charles Hendricks, the then leader of the Lawrence Livermore National Laboratory (LLNL) fabrication effort, recognized the need for this technology exchange in 1981. The series has continued on about an eighteen-month schedule, alternately sponsored by LLNL and Los Alamos National Laboratory. The tenth, sponsored by Los Alamos, continued the tradition as a highly useful technical exchange, but it was unique in that it was the first meeting that was completely unclassified. A dramatic change in the US Department of Energy classification guidance made this change possible.

As a result of the new classification guidance, we were able to invite more foreign participation. In addition to participants from the US, UK and Canada, representatives from France, Japan, and two Russian laboratories attended, about 115 in all.

This booklet of the presentations is as complete as we could make it. Some of the papers contained here may be incomplete; feel free to contact the authors for more information. A list of participants appears after the papers themselves. About fifteen authors accepted the offer to publish more complete versions of their papers in a dedicated issue of Fusion Technology, which should be out in December.

I would like to thank Jean Stark for all her help organizing the conference and assembling this publication. Thanks as well go to all the participants who supplied copies of their overhead transparencies and posters for this book and whose work made the conference worthwhile.

Larry R. Foreman
Technical Host
Tenth Target Fabrication Specialists’ Meeting
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Overview of US Inertial Confinement Fusion Program

presented to
Tenth Target Fabrication Specialists' Meeting
Taos, NM

February 6, 1995

Presented by
George York
Science Advisor (LANL)
Office of Research & Inertial Fusion
U.S. Department of Energy
There are two major, but very different, approaches to laboratory thermonuclear fusion: Inertial Confinement Fusion (ICF) and Magnetic Confinement Fusion (MCF)
The ICF Program mission is to provide a thermonuclear capability in the laboratory for defense and ultimately civilian applications.

The ICF approach is to use a laser or particle beam to compress and heat a small pellet to densities and temperatures similar to those found in thermonuclear weapons and stars, and generate fusion yields many times the input beam energy.

- Weapons physics and weapons effects
- Maintain nuclear weapons competence (particularly in the event of reduced testing)
- Electrical energy production in the 21st century

The ICF concept is to produce, in a laboratory environment, a short lived, miniature explosion which will release energy by thermonuclear fusion.

- Heating of the Ablator
  - uniform irradiation with a laser, particle beam, or x-ray source rapidly heats the outer surface (ablator) of the spheroidal fuel capsule
- Ablation and Compression
  - outward streaming ablation material produces an inward directed, rocket-like force that compresses the DT fuel
- Compression and Heating
  - careful tailoring of the implosion produces compression and heating of the DT to simultaneously produce a central hotspot with \( p = 100-500 \) x solid density having \( T_D > 5 \text{ keV} \) and a main fuel region of \( p = 1800-6000 \) x solid density at low temperature
- Ignition and Thermonuclear Burn
  - hotspot ignition and propagating burn through the compressed main fuel region produces fusion energy yield many times the input driver energy
Direct- and indirect-drive ICF are being pursued

A multi-element, multi-participant, effort is essential for a successful ICF program

**Driver Development**

- Glass laser (Indirect drive) - LLNL
- Glass laser (Direct drive) - University of Rochester/LLE
- Gas laser (KrF) - Naval Research Laboratory
- Light ions - Sandia National Laboratory
- Heavy ions - OER/LBL

**Target Fabrication**

- LANL/LLNL/General Atomics
- Cryogenic targets & handling - UR/General Atomics

**Target Physics**

- High gain - LLNL/LANL
- Laboratory indirect drive - LLNL/LANL/SNL
- Laboratory direct drive - LLNL/UR/NRL
The entire ICF program was reviewed by the National Academy of Sciences in 1990

Major Recommendations & Status

Highest Priority is to demonstrate Ignition & Gain

Build Precision Nova and resolve target issues

*Precision Nova was successfully completed and used for Nova technical contract target physics campaign. Nova technical contract nearly complete thanks to the collaborative efforts of LLNL and LANL. Successful NIF target design completed.*

Validate National Ignition Facility laser architecture

*Prototype Beamlet successfully built and tested (>5kJ @3ns, 3ω)*

*Conceptual Design for NIF completed*

*KD-1 approved October 21, 1994*

Build Omega Upgrade

*Omega upgrade to commence operations April 1995*

Restore Inertial Fusion to Office Status in DASMA and establish an Inertial Confinement Fusion Advisory Committee

*Office of Research and Inertial Fusion (Marshall Sluyter, Director) reports to Dr. Roger Fisher, DASRAD. ICFAC established as a Federal Advisory Committee reporting to ASDP. ICFAC has held six reviews of various aspects of the program and continues in an ongoing status.*

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The entire ICF program was reviewed by the National Academy of Sciences in 1990

Major Recommendations & Status

Accelerate construction of the Nike KrF laser at NRL

*Nike laser completed in October 1994, now commencing operations.*

Level-of-effort for light ion program - review in two years

*ICFAC review held March 1993. Significant progress reported.*

Inertial Fusion should remain primarily a Defense Program

*ORIF reports directly to DASRAD within DP. IFE activities moved to OFE.*

Form a panel to review ICF classification guidelines

*Some declassification has occurred - final report awaiting HQ approval.*
The Inertial Fusion Advisory Committee (ICFAC) advises DOE on technical and management aspects of the ICF program

- Ongoing Federal Advisory Committee to advise the ASDP on program plan and strategies, recommended changes, pace and scope of the ICF program, specific technical issues, and new facilities and upgrades. The following issues have been reviewed:

  - **Time-dependent hohlraum asymmetry for indirect-drive ICF**
  - **Light ion program**
  - **KrF laser program**
  - **Nova Technical Coi**
  - **NIF KD1 readiness & the UR/LLE program**
  - **Program priorities & Importance of ICF to SBSS**

United States Department of Energy Inertial Fusion Program

 Secretary
 Deputy Secretary
 Under Secretary

- Office of Energy Research
- Office of Fusion Energy
- Applied Plasma Physics Division

Office of the National Ignition Facility

Office of Research & Inertial Fusion

- SNL
  - L'att Ions
  - Inc. rect drive
- LLNL
  - Glass Laser
  - NIF Indirect drive
- LANL
  - Target Issues
- UR/LLE
  - Glass Laser
  - Indirect drive
- NRL
  - KrF Laser
  - Direct drive
- General
  - Atomics
  - Targets
- LBL
  - Heavy Ions
ICF milestones and decision points
ICF Program Participant
Lawrence Livermore National laboratory

- Program emphasizes indirect drive with Nd:glass laser
- Target physics program, as described by the Nova Technical Contract, is aimed at establishing the technical basis for ignition and gain with indirect drive
- Specifications for hohlraum energetics, symmetry, Rayleigh-Taylor growth, laser plasma interaction, pulse shaping, and ignition requirements are all consistent with experiments and modeling
- Nova Technical Contract experiments are being extended to answer new questions (such as gas filled hohlraums)
- Beamlet, a scientific prototype of a NIF beamline has achieved >7 KJ at 3 ω, 3 ns
- The NIF laser design at 1.8 MJ/500 TW has margin to cover uncertainties
- The NIF can be configured for both indirect and direct drive
ICF Program Driver
Glass Lasers

- U. of Rochester/Laboratory for User Energetics effort emphasizes direct drive with Nd:glass laser

- Past experiments with 2 kJ OMEGA laser demonstrated:
  - smoothing by spectral dispersion (SSD) of individual beams
  - 4.6% rms irradiation nonuniformity with 24 beams
  - 100 - 300 x liquid DT density measured target compressions

- OMEGA Upgrade laser (30 kJ @ 0.35 μm, 60 beams), presently under construction. Activation imminent
  - Will investigate ignition scaling
  - 1-2% drive uniformity predicted

ICF Program Participant:
Los Alamos National Laboratory

- Independent design capability to determine performance of targets and address physics issues for both direct and indirect drive

- Major participant in target experimental campaigns for Nova Technical Contract

- Actively contributing to NIF laser activities and target designs

- Applying ICF science and technology to weapons physics issues

- Pursuing advanced target and target handling concepts

- Full range of target production capability

- Operates Trident glass laser for diagnostic development and other high energy density experiments
ICF Program Participant
Sandia National Laboratories

- Program emphasizes indirect drive with light ion beams
  Center for Pulsed power development

- Particle Beam Fusion Accelerator-II (PBFA-II) past
  experiments have produced:
  - <20 mrad divergence with lithium beam
  - 2.5 TW/cm² focal intensity with 9 MeV-lithium beam
  - over 60 eV hohlraum temperatures

- Surface cleaning on SABRE (linear beam) increased
  lithium beam output

- PBFA-II undergoing modification to reduce impurity
  induced parasitic losses
  - pre-shot glow discharge cleaning
  - titanium getter added
  - experiments imminent

ICF Program Participant:
Naval Research Laboratory

- Program emphasizes direct drive with KrF gas laser

- Inherent spatial incoherence (ISI) beam smoothing technique demonstrated

- Expected to deliver 2-3 kJ in 4 ns, 248 nm pulse from 44 beams - 12 beams
  (800J) used for back and side lighters

- CCD camera measurements of output from the 20 cm amplifier show time
  averaged intensity fluctuations of < 1.3% within central 65% of beam diameter

- Measurements agree well with model

- Measurements of focal profiles and energy from final amplifier are currently
  in progress
The Justification of Mission Need for the NIF was established by the Secretary of Energy in January 1993 (Key Decision 0)

- A DOE project charter was signed, and conceptual design funding was provided, in April 1993
- A multi-laboratory project organization was formed, and a memorandum of agreement was signed, in August 1993
- The Conceptual Design Report (CDR) was completed in May 1994
- The Project has been validated by independent review groups and by DOE
  - scientific basis - Inertial Confinement Fusion Advisory Committee (ICFAC)/DP Office of ICF
  - cost estimate - Foster-Wheeler USA Corporation/DOE Office of Field Management
  - project readiness - Independent contractors/DP Office of Construction and Capital Projects
- Approval for New Start and Title 1 (Key Decision 1)
  October 20, 1994

Primary Criteria for the National Ignition Facility have been proposed by the DOE Defense Program's laboratories

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<td>Laser pulse wavelength</td>
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<tr>
<td>Beamlet power balance</td>
<td>&lt; 8 % rms over 2 ns</td>
</tr>
<tr>
<td>Beamlet pointing accuracy</td>
<td>&lt; 50 μm</td>
</tr>
<tr>
<td>ICF target compatibility</td>
<td>Cryogenic and non-cryogenic</td>
</tr>
<tr>
<td>Annual number of shots with</td>
<td>100 with yield 1 kJ - 100 kJ</td>
</tr>
<tr>
<td>fusion yield</td>
<td>35 with yield 100 kJ - 5 MJ</td>
</tr>
<tr>
<td>Maximum credible DT fusion yield</td>
<td>10 with yield 5 MJ - 20 MJ</td>
</tr>
<tr>
<td>Classification level of experiments</td>
<td>45 MJ</td>
</tr>
<tr>
<td></td>
<td>Classified &amp; unclassified</td>
</tr>
</tbody>
</table>
Target ignition regime is an area in power-energy space

Operating regime constrained by laser-plasma instabilities and hydrodynamic instabilities

Margin above threshold provides room to tradeoff asymmetry, laser-plasma instabilities, other uncertainties

NIF Mission Statement (from Functional Requirements/Primary Criteria)

The National Ignition Facility (NIF), proposed by the Office of Inertial Confinement Fusion (ICF), will be a key element in the Defense Programs aboveground experimental (AGEX) capabilities for maintaining nuclear competence and weapons effects simulation. The primary mission of the NIF, after construction and activation activities are completed, will be to demonstrate fusion ignition in the laboratory. The most immediate application of the NIF will be to provide nuclear-weapon-related physics data, since many phenomena occurring on the laboratory scale are similar and relevant to those occurring in weapons. The NIF may also provide an important capability for weapons effects simulation. The second purpose for the NIF is to achieve propagating fusion burn and modest energy gain for development as a source of civilian energy, as stated in the NIF Justification of Mission Need which was endorsed by the Secretary of Energy, the DOE's Fusion Policy Advisory Committee, and the National Academy of Sciences Inertial Fusion Review Group.
The National Ignition Facility

National Ignition Facility Target Area
The Beamlet laser is a scientific prototype of an NIF beamline

<table>
<thead>
<tr>
<th>Amplifier configuration (cav A1-A2-booster A3)</th>
<th>Beamlet 11-0-5</th>
<th>NIF 9-5-5</th>
</tr>
</thead>
<tbody>
<tr>
<td>High fluence (equivalent) pulse duration</td>
<td>13.2 J/cm² (5.0 ns)</td>
<td>13.3 J/cm² (5.1 ns)</td>
</tr>
<tr>
<td>Beam area (half power points)</td>
<td>1027 cm²</td>
<td>1280 cm²</td>
</tr>
<tr>
<td>Near field fluence modulation (pk-to-avg)</td>
<td>1.25–1.35</td>
<td>1.5</td>
</tr>
<tr>
<td>Spatial gain compensation contrast</td>
<td>3:1</td>
<td>5:1</td>
</tr>
<tr>
<td>Beam divergence:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wavefront precorrection</td>
<td>±20 μrad</td>
<td>±35 μrad</td>
</tr>
<tr>
<td>Static correction only</td>
<td>±35 μrad</td>
<td></td>
</tr>
<tr>
<td>Phase modulation bandwidth</td>
<td>30 GHz</td>
<td>30 GHz</td>
</tr>
<tr>
<td>Cavity optical switch efficiency</td>
<td>89.6%</td>
<td>99.5%</td>
</tr>
<tr>
<td>High fluence (equivalent) pulse duration</td>
<td>7.7 J/cm² (3.0 ns)</td>
<td>8.0 J/cm² (3.6 ns)</td>
</tr>
<tr>
<td>3 α conversion efficiency</td>
<td>81% average “square pulse”</td>
<td>85% peak “shaped pulse”</td>
</tr>
</tbody>
</table>

Near-term demonstration of advanced solid state laser multipass architecture is proceeding as part of LCF baseline technology program.

Performance milestone of >5kJ in 3ns at 0.35μm on schedule for FY94.
The OMEGA Upgrade project baseline schedule

<table>
<thead>
<tr>
<th>DOE-HQ Milestones</th>
<th>Project Office</th>
<th>Design</th>
<th>Procurement Contracts</th>
<th>In-House Manufacturing</th>
<th>System Integration</th>
<th>Funding Profile</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 2 3 4</td>
<td>1 2 3 4</td>
<td>1 2 3 4</td>
<td>1 2 3 4</td>
<td>1 2 3 4</td>
<td>1 2 3 4</td>
<td>$8.76M</td>
</tr>
<tr>
<td>KU2 Start Title II Design</td>
<td>KU5 Start Title III Construction</td>
<td>PMF Approved</td>
<td>KU5 Design Complete WBS 2.0 &amp; 3.0</td>
<td>KU6 Transition to Operations</td>
<td>Operations Readiness Review</td>
<td>$20.3M</td>
</tr>
<tr>
<td>FOHSI Approved</td>
<td>PMF Approved</td>
<td>Title II Design Complete</td>
<td>Title II Design Complete</td>
<td>Acceptance Test Plan Submitted</td>
<td>Laser Hardware Delivered</td>
<td>$22.14M</td>
</tr>
<tr>
<td>EA Submitted</td>
<td>Complete Shielding Design</td>
<td>Start Long Lead Items</td>
<td>Deliver Target Chamber</td>
<td>Start Optics &amp; Laser Amplifier Mfg. &amp; Ass'y</td>
<td>Optics &amp; Laser Amplifiers Assembled</td>
<td>$8.0M</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Install Target Chamber</td>
<td>Compute Initial Target Shots @ 80 Hz, 90%-75% rms</td>
<td>$1.8M</td>
</tr>
</tbody>
</table>

OMEGA Upgrade Laser Integration is nearing completion and Activation is in-process

- All (104) Laser and Target Structures are installed and aligned.
- Main & Foot-Pulse Drivers and one "ABC" beamline are activated.
- All Power Conditioning units are installed (42 MJ).
- Rod & Disk Amplifier Installation is 60% complete.
- Laser and target bay optical systems will be complete Dec '94.
- Control Systems are being activated as required.
- Activation and performance testing of first IR beamline will be complete in Oct '94.
- Activation and performance testing of first frequency converted 10-beam cluster is planned for completion in Nov '94.

The OMEGA Upgrade Project is on track for the 2Q95 KD4 Project Complete Milestone.
All major structures are installed in the target bay

The target area is designed for precision alignment and long-term stability

- Alignment-sensor packages are built into Granitan™ structures
- Rigid steel structures support transport mirrors
- Focus lenses are mounted on the target chamber
The Nike KrF Laser Facility

Nike is nearly complete and has achieved many of its technical goals

- demonstrated single-beam focal uniformity of 2% after the final amplifier
- obtained 5 kJ output from final amplifier
- stable overlap of 10 beams on target demonstrated while firing all amplifiers

The laser works reliably enough to allow target experiments

60 cm amplifier has achieved 5 kJ output in 56 beams

solid lines show kinetics calculations vs energy into gas

![Graph showing output laser energy vs input laser energy with markers for 56 kJ into laser gas and 70 kJ into laser gas]
After amplification by the 60 cm amplifier the focal profile remains highly uniform

(3,900 Joules in 56 beams)

Focal uniformity after 60 cm amp., multi-kilojoule shots

25 Jan. data  30 min. between some shots
The main advantage of a KrF laser for ICF is its outstanding beam quality.

Measured focal distributions of single 4 ns beams at the output of the 20 cm amplifier:

- RMS Focal Spot Nonuniformity

- Over 60% of the FWHM diameter: Planar Tilts < 2%, Quadratic Curvatures < 2%
- RMS Speckle Nonuniformities = 0.8%-1.3% (all modes)

**Summary and Status**

- The ICF program has been firmly established as an important element within the stockpile stewardship program by ICFAC and JASON studies.
- The Nova technical contract is nearly complete and results lend substantial confidence to NIF success.
- The NIF conceptual design was completed, KD1 was approved, the Beamlet successfully met its milestones and a project office reporting directly to the Deputy Assistant Secretary has been established.
- Improved design capability and benchmarking have yielded several successful NIF target designs, from both design labs.
- SNL is anticipating 100 eV experiments by 2Q FY95.
- The Nike KrF laser has met its design goals for both energy and uniformity and is beginning target experiments.
- The Omega Upgrade is on schedule for operations commencing in 2Q FY95 with ultimately 30 kJ for direct drive experiments.
The OMEGA Upgrade Experimental Program Will Validate the Direct-Drive Approach

Summary
The OMEGA Upgrade direct-drive program has the central objective of high-gain capsule-performance validation

Experiments planned will examine
- Laser energy coupling and energy transport
- Irradiation uniformity requirements
- Hydrodynamic stability
- Hot-spot/main fuel layer physics
Outline

- Objectives of OMEGA Direct-Drive Experimental Program
- OMEGA laser
- OMEGA Direct-Drive Experimental Plan
  - Energy coupling and transport
  - Laser-plasma instabilities
  - Plasma hydrodynamic experiments
  - Surrogate cryo implosion experiments
  - Cryogenic experiments

Objectives

The mission of the UR/LLE program is to validate direct-drive capsule performance

- Develop required laser and target diagnostics
- Demonstrate 1%-2% (rms) irradiation uniformity
- Demonstrate versatile, high-dynamic-range pulse shaping
- Understand limitations imposed by R-T instabilities
- Understand limitations imposed by laser-matter interaction physics
- Implement hydrodynamically equivalent cryogenic capsules
- Understand key target physics issues and extrapolate to NIF condition
The OMEGA Upgrade program is a multilaboratory effort to validate the direct-drive target approach.

**Theoretical design**
1-D, 2-D hydro codes (LLE)
Hydrostability (LLE, LLNL, LANL)

**Convergence ratio > 20**
G.F. > 500
ρR > 0.2 g/cm
T > 2 keV

**Target diagnostics**
(LLE, LLNL, LANL)

**Target fabrication**
- Cryogenic fuel;
- Polymer shells (LLE, LLNL, GA, LANL)

**Laser capabilities**
OMEGA Upgrade
Uniformity, pulse shaping (LLE, LLNL)

---

The OMEGA laser system provides a unique experimental facility for science-based stockpile.
The main-pulse regen in the pulse generation room has been activated.

The driver line for the OMEGA Upgrade met its April 1994 activation milestone.
The power conditioning for the Vega grade amplifiers is in place.

The 120 disk amplifiers are part of a new V54-HK system designed and built by Maxwell Laboratories Inc.

The S&H structure is designed for serviceability and maintainability of the amplifiers.
Amplifier configurations were engineered for ease, inspection and maintenance.

Target bay structures are complete and installation of optics has begun.
LLE direct-drive program schedule FY95-99 shows completion dates for various program elements.

<table>
<thead>
<tr>
<th>OMEGA Upgrade Construction</th>
<th>Laser Physics Program</th>
<th>Power Balance</th>
<th>Individual Beam Uniformity</th>
<th>Temporal Pulse Shaping</th>
<th>Diagnostic Development Program</th>
<th>Diagnostic Implementation</th>
<th>Target Physics Program</th>
<th>Plasma Physics Experiments</th>
<th>Hydrodynamic Stability Experiments</th>
<th>Hydrodynamic-Equivalent Implosion Experiments</th>
<th>Target Fabrication/Production Program</th>
<th>Polymer Bead Fabrication</th>
<th>Polymer Shell Capsule Fabrication</th>
<th>Surrogate Polymer Capsule Fabrication</th>
<th>Fritt Foil Fabrication</th>
<th>Cryogenic Capsule Fabrication</th>
</tr>
</thead>
<tbody>
<tr>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
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<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
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<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
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<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
<td>FY95 FY96 FY97 FY98 FY99 FY00</td>
</tr>
<tr>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
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<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
<td>1 2 3 4 5 6 7 8 9 10 11 12</td>
</tr>
</tbody>
</table>

A comprehensive experimental program is being planned for the OMEGA Upgrade to address the key physics issues associated with direct-drive capsule implosions.

- Laser energy coupling and energy transport
  - absorption fraction
  - x-ray conversion
  - ablation-rate experiments
  - gas-filled/voided implosion experiments
- Laser-plasma interaction physics
  - long-scale-length plasma experiments
- Irradiation uniformity
  - development program
  - experiments to test modifications/improvements
A comprehensive experimental program is being planned for the OMEGA Upgrade to address the key physics issues associated with direct-drive capsule implosions (continued)

- Hydrodynamic stability
  - planar
  - spherical
- Hot-spot physics
  - noncryogenic "surrogate" HET capsules
  - cryogenic HET capsules

Energy coupling and transport
Direct-drive implosions require understanding and control of laser-plasma interactions

- Short-wavelength irradiation eliminates preheat from resonance absorption fast electrons and results in efficient laser-energy absorption.
- Multiple-beam irradiation allows high driving intensities while remaining below stability thresholds.
Initial experiments planned for the OMEGA Upgrade will use capsules similar to those used on OMEGA.

Diagnostic development and physical experiments

Asorbed laser energy experiments coupled with high-speed x-ray streak photography will be used to confirm capsule drive and symmetry.
The development of imposed $l = 1$ and $l = 3$ modes was examined for direct-drive implosions on OMEGA.

Agreement between experiment and simulation was found for both the zeroth order as well as the perturbed hydrodynamic motion.
Relatively simple "finite mass" planar foils will be required for the long-scale-length experiments.

Planar hydrodynamic experiments
Flat-target experiments are an integrated part of the planned OMEGA Upgrade experimental program.

- The NOVA RT direct-drive results are being used to plan future experiments at the University of Rochester.
- The multiple-beam configuration of the OMEGA Upgrade system will allow the target-illumination effects of beam-beam overlap and steep incident angle to be measured.
- Flat-target hydrodynamic experiments will be used to test advanced uniformity improvements, pulse shaping and Rayleigh-Taylor growth-rate reduction.
- Spherical-implosion data will be compared to the flat-target hydrostability data to test target improvements.
Surrogate cryo implosion experiments

Many of the "all" polymer capsules developed for OMEGA experiments will be used during phases of OMEGA Upgrade experiments.

Transport and drive uniformity

“Hot-spot” performance

Surrogate cryogenic "main fuel" layer performance

- Radii from 350–500 μm
- Total thickness 20–50 μm
- We will want to examine capsules with CH ablators doped with high-Z materials
- We will want to dope DT or D₂ with high-Z materials

Cryogenic capsules

Until experimental data from initial OMEGA Upgrade experiments are obtained, numerical simulations will be used to determine OMEGA Upgrade cryogenic capsule specifications.

1. Surface finish of laser barrier layer and outer surface ≤500 Å rms
2. Inner DT interface — ≤1000 Å
3. Concentricity (neutron yield not severely reduced; however, may influence interpretation of some ρR diagnostics)
   - ≤2.5 μm $P_1$ for DT
   - ≤0.5 μm $P_1$ for CH

Requirements for DT and CH bulk uniformity (voids, cracks, etc.) and opacity variations in high-Z-doped CH ablators are under investigation.
LLE is examining the use of wetted foam cryogenic capsules on the OMEGA Upgrade

- Wetted focus may allow for the construction of thick fuel layers using either DT or D₂.
- Radiochemistry (rad-chem) diagnostics can be used to measure ρR using Si-doped CH foams.
- Use of low-Z wetted CH foams on the OMEGA Upgrade does not have a dramatic impact on capsule performance (in 1-D).
- However, its use, especially with D₂, may have an important impact on diagnostics used to examine final core conditions.

Summary/conclusion

The OMEGA Upgrade direct-drive program has the central objective of high-gain capsule-performance validation

- Experiments planned will examine
  - laser energy coupling and energy transport
  - irradiation uniformity requirements
  - hydrodynamic stability
  - hot-spot/main fuel layer physics

Target fabrication will play an increasingly important role as the OMEGA Upgrade experimental program progresses.
Status of the Nike KrF Laser Facility

Presented by John Sethian
Naval Research Laboratory

Co Authors
Naval Research Laboratory, Washington, DC

A.V. Deniz, J. Hardgrove and T. Lehecka
Science Applications International Corporation

W.D. Webster
East Coast Engineering

M.W. McGeoch
PLEX Corporation
NIKE is being developed to investigate the physics of direct drive Inertial Confinement Fusion (ICF)

Nike will use flat foil targets of the same thickness (mass) as a high gain ICF target, with the same laser intensity as a high gain system.

Flat foil represents one sector of high gain target

Flat foils allow full diagnostic access

The Primary Advantage of a KrF laser is the outstanding beam quality

Nike Experimental Beam Focal Distribution

Single beam fluence:
- Tilt: 1.9% 
- RMS: <1.6%

Predicted with 44 overlapped beams:
- Tilt: <1.0% 
- RMS: 0.25%

<= Fluence in single beam
Total of 120 J in 28 beams
~ 4 ns/beam

38
Improved laser uniformity and target smoothness may lead to a very high pellet gain.

\[
\text{Gain} = \frac{\text{energy out}}{\text{energy in}}
\]

KrF lasers can produce high quality beams because they allow low distortion and rapid smoothing by \textit{Induced Spatial Incoherence (ISI)}

The large bandwidth of KrF (> 2 THz) allows short averaging times, hence rapid smoothing

The low linear (and nonlinear) phase aberration of KrF allows minimal profile distortion

KrF lasers can also be operated with unpolarized light, which would give an additional \(\sqrt{2}\) smoothing factor for nonuniformities at all spatial frequencies
The Nike Laser consists of a broadband incoherent source that is imaged through the amplifiers and focusing lenses onto target.

The 60cm amplifier has achieved 5 kJ in 56 beams.

Solid line shows kinetics calculation for 50 KJ input from 20 cm amplifier.

Assume 20% lost to ASE; 10% to radiation, and allow for laser.
The Nike Laser not only has the required energy, but the required highly uniform focal profile as well.

After amplification by the 60 cm amplifier; 3900 Joules in 56 beams

The focal plane nonuniformity after the 60 cm amplifier is quite low:

(Planar fit over center half of FWHM diameter)

RMS deviation: 1%, tilt: 0.8%, peak to valley deviation: 2.3%
Nike has been fired twice per hour producing beams at the multikilojoule level with good focal uniformity.

January 25; five shots, approx. 30 minutes between each shot

The NIKE Target Chamber will feature 44 beams for target illumination and 12 beams for an x-ray backlighter.
NIKE ablative-acceleration experiments will utilize planar targets.

D>2d to obtain "planar" acceleration

target parameters  
laser parameters  
3 to 12 mg/cm^2 areal density  
1 = 1 to 3 x 10^{14} W/cm^2  
distance accelerated (d) = 0.1 to 0.3 mm  
focal diameter (D) = 0.5 to 0.8 mm  
final velocity 100 to 200 km/sec  
E = 2.2 kJ in 4 nsec

Targets will be varied to study and control RT instability and preheat

TYPE I: Laser/Diagnostic setup + first Rayleigh-Taylor Exp

TYPE II: Early-time X-Ray source to reduce imprinting

TYPE III: Control Rayleigh-Taylor and not preheat fuel
Very uniform lasers require very flat targets.

First set of targets are flat CH foils:

- thickness 20 to 100 μm (ACHIEVED)
- FLATNESS: 1 μm/mm (ACHIEVED < 1)
- surface finish < 100 Å (peak-valley) (ACHIEVED < 20 Å)

These targets have been fabricated for us by WJSA. Thanks to:
Chuck Hendricks
Brendan Casey
Brian Motta
Steve Dropinski

Summary

Goal of Nike program is to study direct drive using a very uniform laser
High uniformity may lead to very high gain ICF

The Nike laser has shown engineering/scientific feasibility of its stated goals:
- 4 kJ laser energy
- < 2% nonuniformity
- 2 shots per hour

Long term reliability still needs to be demonstrated

Targets have been made with surface finishes that match the laser uniformity

Expect serious target experiments to start within 1 month
Some target experiments have already begun
General Atomics has been producing composite polymer capsules for the national ICF program for the past two years. The process is based on the process developed at Lawrence Livermore National Laboratory. Several innovations make the process more reliable. This paper will present an overview of the GA process, noting recent innovations.

The process starts with the production of polymer shells, prepared by injecting droplets of polystyrene solution into a vertical tube heated to about 200°C. The shells are sorted by sieving to a tight diameter range and then selected by optical microscopy. The shells are coated with PVA by dropping a shell covered with an aqueous solution of PVA into another heated vertical tube that evaporates the water and deposits the PVA on the outside surface of the shell. The yield from this historically low yield step was greatly improved by using an improved injection device. PVA-coated shells are carefully selected and then coated with CH polymer by the glow discharge polymerization (GDP) technique. Finally these capsules are filled with diagnostic gas(es) by permeation. Permeation after GDP coating is a recent innovation that reduces the shell to shell pressure variation compared to the previous method where the shells were filled just before coating with PVA. Each capsule is carefully characterized for ID, total wall, and layer thicknesses (by radiography), $\Delta_{wall}$ (by interferometry), surface features (by optical microscopy), and for argon content (by XRF). Many of the characterization steps are computerized.

Innovations in the process have made it easier to make capsules meeting difficult requirements. Among these are the “exacts”, the “symcaps”, capsules with deuterated shells, and capsules with germanium-doped GDP.
COMPOSITE POLYMER CAPSULE PRODUCTION AT GENERAL ATOMICS

W. MILLER
J. ANKNEY
W. BAUGH
D. BEIGHLEY
L. BROWN
J. GIBSON
M. HOPPE
D. HUSBAND
D. STEINMAN
R. STEPHENS

10th TARGET FABRICATION SPECIALISTS' MEETING
TAOS, NEW MEXICO

February 6-10, 1995
ICF CAPSULES MUST MEET STRINGENT SPECIFICATIONS

Polystyrene shell
(doped with D, Cl, Ti)
-440 μm i.d., 2-4 μm wall

PVA
2-4 μm

Diagnostic gas
(Ar, Ne, Xe)

CH (by GDP) 15 - 60 μm
(doped with Ge, Cl)
surface finish ≤ 1000 Å RMS

Δw ≤ 10%

GA ADAPTED AND MODIFIED THE LLNL PROCESS

PS

PVA

GDP

Permeation

Optical

Radiography

XRF
DOPED POLYMER DECREASES SHELL QUALITY

1.000K

Ti-(OC₃H₇)₃

1-X (0.75 < x < 0.85)

Ti polymer

THE YIELD IS INCREASING FROM THE PVA COATING STEP

Precision sizing shells through ±2 μm sieves

Capillary

Squeezing the bulb forces the warm PVA solution into the shell-filled capillary

Squeezing the bulb forces the shells and PVA solution into the drop tower

FY 93 Oct 93-Mar 94 Jul-Sep 94

1.9% 2.5% 3.6%

Good / Injected
Glow Discharge Polymerization

DOPANTS
- Germanium
- Deuterium
- Chlorine

THE ABILITY TO PERMEATE ARGON THROUGH PVA MADE OUR INVENTORY PROCESS POSSIBLE

- Permeation Apparatus
  - Off-the-shelf fittings
  - 60 atm fill capability
- GDP supports the PS mandrel
- Wall uniformity does not suffer
- Each capsule filled to the identical pressure
- Fill pressures greater than 1 atm are possible
- Pressure or gas can be changed later
OUR NEW PRODUCTION PROCESS IS SIMPLE

New Production Process

Take Shells from Inventory → GDP Coat → Argon Fill → Characterize

- no → OK
- yes → Ship

CHARACTERIZATION REQUIRES MANY TECHNIQUES

Optical Microscopy Interferometry
Radiography
XRF

Surface finish (bumps)
Total wall thickness
Concentricity
Diameter
Circularity
Gas pressure

Inner diameter
Outer diameter
Layer thickness
Layer thickness uniformity
Circularity

Diagnostic gas pressure
Mandrel dopants
GDP dopants

Characterization measurements are performed accurately and quickly with a PC.
THE "EXACTS" CAPSULES WERE ESPECIALLY CHALLENGING

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Normal</th>
<th>Exacts</th>
</tr>
</thead>
<tbody>
<tr>
<td>∆ I.D.</td>
<td>± 20.0</td>
<td>± 2.5</td>
</tr>
<tr>
<td>∆ Inner wall</td>
<td>± 3.0</td>
<td>± 0.25</td>
</tr>
<tr>
<td>∆ CH (GDP)</td>
<td>± 5.0</td>
<td>± 0.5</td>
</tr>
</tbody>
</table>

"SYMCAPS" REQUIRED INNER DIAMETERS MATCHED TO 4 µm

○ Symcap A: Total wall = 35 µm
□ Symcap B: Total wall = 20 µm
△ Symcap C: Total wall = 15 µm
WE MADE CAPSULES USING 2 TYPES OF D-DOPED MANDRELS

The capsules made with the microencapsulation-prepared mandrels had better wall uniformity (Δw = 1 μm vs. 3.2 μm) and were more spherical, but had vacuoles.

THE INNOVATIONS IN THE PROCESS HAVE MADE IT EASIER TO MAKE CAPSULES MEETING DIFFICULT REQUIREMENTS

FY95 Deliveries
13 shipments — 199 capsules

- 10 atm Neon
- 2% Ge
- 1.3% Ge
- D-PS 1.3% Ge
- 10 atm Xenon

[Graph showing FY95 deliveries with 13 shipments totaling 199 capsules, with different atmospheres and Ge concentrations marked]
Controlled-mass microencapsulation (CMM) is one technique used to produce highly spherical polymer shells with uniform wall thicknesses for use as Inertial Confinement Fusion (ICF) targets. The CMM process consists of forming a water-oil-water emulsion shell through the use of a triple orifice droplet generator. When a shell is formed in a heated bath, solvent from the oil phase (polymer/solvent solution) diffuses from the shell wall into the surrounding water phases. We have recently developed production techniques and processing conditions which can achieve the strict specifications required of ICF targets. Achieving batch reproducibility and high yield is now our main focus.

The inconsistent appearance of vacuoles and dimpling are the two major defects which must be eliminated. Vacuoles (voids) are believed to be the result of the microphase separation caused by trace amounts of water which diffuse into the shell wall during the solidification process. Computer simulations are being employed to guide the experimental identification of a stable processing window in which the composition trajectory of the wall fluid avoids residence time within the two-phase immiscibility envelope.

Dimples are believed to be caused by osmotic forces on the fragile shell after solidification in the CMM process and also during the subsequent alcohol-driven water extraction steps. We are investigating the use of temperature ramps near the end of the CMM runs and carefully metered alcohol-water extraction to minimize osmotic forces and suppress dimple formation.
PRODUCTION OF LARGE POLYMER SHELLS
VIA CONTROLLED-MASS MICROENCAPSULATION

TENTH TARGET FABRICATION SPECIALISTS' MEETING
February 6 - 10, 1995

Lisa Cheung, Travis Boone, Eben Lilley, & Don Nelson
Soane Technologies, Inc.
3916 Trust Way
Havward, CA 94545

INTRODUCTION

GOAL: To produce target quality, monodisperse, polystyrene shells in the 1 - 2 mm size range with wall thicknesses between 5 and 15 μm.

SPECIFICATIONS:

<table>
<thead>
<tr>
<th>Dimensional</th>
<th>Geometrical</th>
<th>Defect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>Non-Conc.</td>
<td>Vacuoles</td>
</tr>
<tr>
<td>1 - 2 mm</td>
<td>&lt; 5%</td>
<td>0 &gt; 5 μm</td>
</tr>
<tr>
<td>Wall Thick.</td>
<td>Sphericity</td>
<td>Debris</td>
</tr>
<tr>
<td>5 - 15 μm</td>
<td>&gt; 98%</td>
<td>0 &gt; 1 μm</td>
</tr>
</tbody>
</table>

CONTROLLED-MASS PRODUCTION SYSTEM

A serial approach has been used to achieve the target specifications.

1. Dimensional Controlled-Mass Microencapsulation
2. Geometrical Sphericity and Concentricity
3. Defect Vacuoles, Debris
CONTROLLED-MASS MICROENCAPSULATION

Outside Diameter
Controlled by
Before Stripping Flow
Now 3rd Orifice Inside Diameter

Wall Thickness
Controlled by relative flow rates of 1st (Water) and 2nd (Organic) orifices

SPHERICITY AND CONCENTRICITY

Sphericity
Rotary Agitation
Surface Tension

Concentricity
Density Matching - Combined Modeling and Experimentation
Rotary Agitation

DENSITY MATCHING - MODEL VERSUS EXPERIMENTS
Dichloroethane/Toluene at 65 °C
DENSITY MATCHING - MODEL VERSUS EXPERIMENTS

Time ~ 300 Seconds

INTERFERENCE PHOTOGRAPHS OF SHELLS

VACUOLE REDUCTION

Chemistry
- Solvent Selection: Toluene and Dichloroethane
- Solvent Purity: Anhydrous

Processing
- Cleanliness: Filtered 2X through 0.2 microns
- Wall Thickness: As thin as possible, but maintaining mechanical strength
- Shell Number Density: ~ 50 shells per 200 ml water
- Syringe Material: Glass, contamination by plastic

Modeling
- Phase Equilibria: Work with Wilemski of LLNL

VACUOLE ABATEMENT

- >5 μm Vacuoles: April 1994
- ~2 μm Vacuoles: August 1994
- 0 Vacuoles: September 1994
DIMPLE FORMATION

When processing high aspect ratio shells at 1.5 mm diameter, there is a tendency for form dimples.

- Dimples result from osmotic pressure difference between the outer and inner water phases.
- Water is unable to replace the organic extracted from the shell core.

PREVENTION OF DIMPLE FORMATION

- Allow water to replace extracted organic by retarding organic transport.
- Reduce driving force for organic transport by saturating outer water phase with organic.

CURRENT PRODUCT QUALITY

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>SPECIFICATION</th>
<th>AVERAGE</th>
<th>BEST</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>1 - 1.25 mm</td>
<td>1.4 mm</td>
<td>1.38 mm</td>
</tr>
<tr>
<td>Wall Thickness</td>
<td>5 - 15 μm</td>
<td>11 μm</td>
<td>7 μm</td>
</tr>
<tr>
<td>Wmax - Wmin &lt; 5%</td>
<td>Wavg</td>
<td>&lt;5 %</td>
<td>0 %</td>
</tr>
<tr>
<td>Sphericity</td>
<td>&gt; 98 %</td>
<td>&gt; 98.5%</td>
<td>&gt; 99 %</td>
</tr>
<tr>
<td>Vacuoles</td>
<td>0 &gt; 5 μm</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2 μm &gt; 20 &gt; 5 μm</td>
<td>10 - 20</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Debrils</td>
<td>0 &gt; 1 μm</td>
<td>10 's</td>
<td>Several</td>
</tr>
</tbody>
</table>

RECOMMENDED FUTURE WORK

1. Obtain and implement different capillaries to increase the size range of shells.
2. Improve the cleanliness of post-processing to remove debris.
   (a) chemical selection
   (b) process control
   (c) laminar flow hood
3. Improve reproducibility of vacuole free runs
   (a) experiments
   (b) mathematical modeling of mass transfer and phase equilibria
4. Eliminate Dimples
   (a) determine the minimum wall thickness which yields a structurally sound shell
   (b) use processing techniques to support the shell during drying
During the drying of thin (few μm) polymer layers, vacuole formation is believed to occur as a result of phase separation. To better understand and control this process, we are using a multicomponent diffusion formalism to predict compositional changes in the layer as organic solvents diffuse out and water diffuses into the layer. Formation of thermodynamically unstable compositions leads to phase separation. We are using statistical mechanics, the UNIFAP methodology, and empirical data to deduce the required values of transport coefficients and equilibrium phase compositions. Results for several different polymer-solvent combinations will be presented.
Prediction of Phase Separation During the Drying of Polymer Shells

Gerald Wilemski, LLNL
Travis Boone, STI
Lisa Cheung, STI
Don Nelson, STI
Robert Cook, LLNL

Presented at:
Tenth Target Fabrication Specialists' Meeting
Taos, NM
February 6-9, 1995
Large plastic shells are needed for both Omega and NIF targets.

- Microencapsulation is one method for producing these shells.
- Shells must have high sphericity, concentricity, smoothness, and uniformity.
- Most of these objectives appear to be reachable.
- Vacuole formation affects uniformity but is poorly understood.

Solvent removal from the shell wall by diffusion and convection results in a solid polymer shell.
The solubility of water in toluene versus temperature is modeled using a modified form of UNIFAP.

![Graph showing the solubility of water in toluene versus temperature.]

The solubility of water in dichloroethane versus temperature is modeled using a modified form of UNIFAP.

![Graph showing the solubility of water in dichloroethane versus temperature.]
The solubility of water in mixtures of toluene/1,2 dichloroethane/polystyrene is predicted using a modified form of UNIFAP.

Vacuoles are believed to occur as a result of phase separation during drying.

- Initially, water diffuses rapidly into the organic fluid shell reaching saturation.
- As organic solvent leaves, polymer concentration rises, slowing diffusion.
- Driving force for water removal is never high.
- As organic solvent level continues to drop, water is trapped and becomes supersaturated.
- Phase separation into water-rich and polymer-rich phases can then occur.
Prior "engineering" model has been replaced by a fundamental multicomponent diffusion model.

- The old model was very useful in understanding the transient behavior of the shell density, dimensions, and composition.
- It lacks the necessary physics for predicting aqueous phase formation.
- A more fundamental treatment is required.

The multicomponent diffusion treatment overcomes key limitations of the prior model.

<table>
<thead>
<tr>
<th>Prior model</th>
<th>Multicomponent diffusion model</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diffusion treated as superposition of decoupled fluxes.</td>
<td>Mass flows depend on all independent concentration gradients.</td>
</tr>
<tr>
<td>Diffusion coefficients only depend on polymer concentration.</td>
<td>Mutual diffusion coefficients depend on full fluid composition.</td>
</tr>
<tr>
<td>Nonideal solution effects not included.</td>
<td>Nonideal solution thermodynamics directly affects diffusion.</td>
</tr>
<tr>
<td>Limited miscibility of water not treated properly.</td>
<td>Nonideal solution thermodynamics determines water miscibility.</td>
</tr>
</tbody>
</table>
The multicomponent diffusion formalism is derived from linear, irreversible thermodynamics.

- Continuity equation for species $i$:
  \[
  \frac{\partial \rho_i}{\partial t} = -\nabla \cdot \left( \mathbf{J}_i + \rho_i \mathbf{u} \right)
  \]
  ($\rho = $ mass concentration, $\mathbf{u} = $ mass average velocity)

- $N-1$ independent diffusion fluxes $\mathbf{J}_i$
  \[
  \mathbf{J}_i = -\sum_{j=1}^{N-1} D_{ij} \nabla \rho_j
  \]

- $D_{ij}$ related to Onsager phenomenological coefficients $L_{ij}$
  \[
  D_{ij} = \frac{1}{T} \sum_{k=1}^{N} L_{ik} \left( \frac{\partial \mu_k}{\partial \rho_j} \right)
  \]
  ($\mu = $ chemical potential, $T = $ temperature)

- Evaluate $\frac{\partial \mu_i}{\partial \rho_j}$ from free energy of mixing (UNIFAP).

The $L_{ij}$ can be expressed in terms of measurable quantities using fundamental statistical mechanics.

- With one approximation, linear response theory allows us to determine the $L_{ij}$ from time correlation functions. We find:
  \[
  L_{ij} = \left( \frac{\rho}{R} \right) w_i w_j \left[ \frac{\nu}{w_i} D_{ij} - \sum_{k} w_k \cdot D_k \right]
  \]
  where
  - $\rho$ is the density
  - $w_i$ is the mass fraction of component $i$
  - $w_i$ is the molecular weight of component
  - $D_{ij}$ is the tracer diffusion constant of component $i$
  - $R$ is the gas constant

- The tracer diffusion constants, $D_i$, are functions of composition.

- We determine the $D_i$ using the Fujita-Doolittle equation for the small molecules and using reptation theory results for the polymer.
The shell composition trajectory reaches the water saturation curve at 200s; the supersaturation then slowly increases.

Initial composition (mass %):
36.9% Toluene
52.5% 1,2-Dichloroethane
10.6% Polystyrene

The water concentration profiles and shell thickness change rapidly with time.
The shell is initially undersaturated with water, but at later times the supersaturation is slowly increasing.

The multicomponent diffusion model provides results supporting the hypothesis that vacuoles result from aqueous phase separation during shell drying.

- Once the organic shell fluid is saturated with water, supersaturation can occur by a combination of factors:
  (i) Low driving force for removal of water
  (ii) Slowing down of diffusion as polymer concentration rises
  (iii) Continued rapid removal of organic solvents by convective mass transfer

- High water supersaturation can lead to the formation of polymer-free, water-rich regions - vacuoles

- Saturation of the external water phase may help suppress aqueous phase separation in the shell by slowing down the removal rate of organic solvents, thereby allowing the slow diffusion of water to keep up with solvent removal. Water would then not supersaturate in the shell, and phase separation would not occur unless hydrophilic condensation sites were present.
A new generation of laser ICF installations such as the proposed NIF make high and severe demands for capsule targets, central among them that their diameter must be at least 2 mm. The current drop tower technologies of laser target fabrication, used in many laboratories all over the world, unfortunately have a basic limitation, namely that it is impossible to manufacture high quality plastic or glass shells with diameters greater than 1 mm. We have carried out many investigations and now propose a new technology to overcome that limitation which we expect will permit us to manufacture high quality shells more than 2 mm in diameter. At LPI this technology is being realized in a new installation which we call a “Ballistic Furnace”. The installation is in the construction and test stage now. In this report we discuss the foundations of this technology, present the technical design of the installation, explain the operation of the individual units, and present the results of our first tests.
"Ballistic Furnace" - one of the ways to large hollow shells for ICF.

P.N. Lebedev Physical Institute, Russian Academy of Sciences
Moscow, Russia.

A new generation of laser ICF installations such as proposed NIF make high and severe demands for capsule targets, central among them that their diameter must be at least 2 mm. The current drop tower technologies of laser target fabrication, used in many laboratories all over the world, unfortunately have a basic limitation, namely that it is impossible to manufacture high quality plastic or glass shells with diameter greater than 1 mm. We have carried out many investigations and now propose a new technology to overcome that limitation which we expect will permit us to manufacture high quality shells more than 2 mm in diameter. At LPI this technology is being realized in a new installation which we call a "Ballistic Furnace". The installation is in the construction and test stage now. In this report we discuss the foundation of this technology, present the technical design of the installation, explain the operation of the individual units, and present the results of our first tests.

A new generation of ICF Laser installations (NIF for example) make high and severe demands for capsule targets and central among them that their diameter must be at least 2 mm. Beside that such plastic shells will be used as a part of complex laser targets.

The current technologies of hollow shells for laser targets, based on a drop tower technologies, have a basic limitation. The diameter of high quality shells produced by such technologies at vertical tower furnaces reasonable heights usual is not more than 1 - 1.2 mm or slightly larger, if special measures have been taken. On the other side there are several different methods - microincapsulation, micro-machining and others for large hollow plastic shells producing, but quality of this shells (surface finish, wall homogeneity etc.) is often worse then quality of shells formed during free fall through a hot gas.
At LPI we have carried out many investigations and have propose a new technology to overcome mentioned above size range limitation. Now we discuss essential features and technical design of new installation developed in accordance with our concept.

The main idea - the initial granule must be propelled upwards through the hot gas along a ballistic trajectory. That is why we call our installation the "Ballistic furnace".

We clearly understand many scientific and technical questions must be experimentally answered before we will be able to achieve maximum shell size and quality. That's why we have designed flexible installation not only for producing shells, but mainly for obtaining experimental data concerned with shell formation processes and optimal working parameters.

The principal features of installation are:
- the initial granule propelling upwards along a ballistic trajectory;
- precise and automatically controlled injection of initial granule;
- automatically controlled temperature profile in the hot zone;
- fast cooling of blown shells;
- full computer controlled operation and parameters measuring.
- easy and fast reconfiguration and working parameters changing;

On the schematic diagram of the "Ballistic furnace" (fig.1) the key units have been highlighted by solid lines: a hot zone (2) with separate electrical heaters; an injector and initial granules feeding system (4); a zone of cooling (5); a vacuum slide gate (7); just-ready shells collector (8); pumping (20) and gas supplying system (18); a computer control and parameter measuring system (10-12,14,15,17,19). This units constitute the first phase of the installation and they have been made and are under assembling, testing and adjusting at present.

The additional parts: an optical registration system (1); a fast preheating unit (3); a high rate cooling unit (6) and corresponding equipment for control and synchronization (9,13,21) are shown on fig.1 by dashed lines. This units we are going to incorporate into the installation later after calibration runs and the first results processing. Its design and working parameters are strongly depended on a maximum size and a quality of shells that will be produced in the first experiments. It will be easy to start work with this parts in a short time because of considerable flexibility installation at whole.

Let us consider design and operation of "ballistic furnace" (fig.2) in
more detail. The initial granules are fed by the supplying device (7) from the container to the injector (8) one by one. The feeding rate is controlled by the computer and synchronized with the injector working cycle. The injector propels a single particle upwards when an acceleration pulse applied to the injector coil and after about 1 sec system is ready to start a new cycle. Previously measured injection accuracy is not worse than 2 mrad. The injector with a trajectory adjustment equipment (9) and a granules feeding device are located in a separate zone of installation (10).

Between the injector zone and the hot zone a zone with several windows is placed (6). It provides for initial granules flight and formed shells observation and for optical preheating of granules if it will be necessary.

The formation of hollow shells occur in the hot zone (1), where the initial particle is heated as it move up through the hot gas, begins to foam near the upper point of trajectory and then turns into a hollow shell during free fall in the hot gas. The height of this zone is 1 m, diameter - 200mm. Wire electrical heater (3) is wrapped around a quartz tube (2) and is divided into three independent controlled sections for desirable temperature profile creating. The temperature is measured in several points along the hot zone by a set of thermocouples and runs into about 1000°C. Usual working atmosphere in the furnace is the mixture of He and Ar at pressure between 2 and 10 kPa.

Using described hot zone and ballistic principle we can achieve the same results as could be obtained in a 4-5 m free-fall column. It is possible to increase the height of the "Ballistic Furnace" hot zone three times by setting up the additional sections, but we are strongly restricted to working room height at present.

For the same reason we are forced to use additionally the high rate cooling system. The free-fall cooling zone (12) have a height 1 m. It is enough for cooling shells not more than 1.5 mm. We are going to use fast cooling to avoid shrinkage and possible deformation of large shells. One of the variant is a falling of blown shells through the cold water drop cone formed by a system of atomizers. In this case an intensity of hot shell heat loss will be about ten times more then in a cold gas.

The electrical vacuum slide gate (13) located between the free-fall cooling zone and the shells collector (14) allows to remove blown shells from the installation without depressurization. The "Ballistic Furnace" at
whole is mounted on the heavy and stable platform.

We have paid a large attention on the developmental stage of the "Ballistic Furnace" to achieve repeatable and continuously monitoring conditions for each shell during its making. That is why we have developed electronics and software for full computer control of installation. This system is schematic shown on fig. 3.

The installation control proceeds by use of the set of modules in CAMAC standard and IBM PC/AT computer (1). Modules (3,4,5) forms specified pulses for the injector control. Analog signals from temperature and pressure sensors are switched by multiplexer (7) and are converted to digital one in ADC module (6) and then are processed in computer. There are 23 independent channel for input data now. Module (8) controls independently heaters in hot zone according to preset regulation low. Another modules (9,10) switches vacuum and gas valves. The system status is monitored continuously by status module (11). No special module for synchronization is provided and synchronization is executed by computer.

We hope to solve a number of problems concerned with laser target fabrication by the new installation using:
- production of high quality plastic shells up to 3 mm in diameter;
- direct observation of shell formation processes;
- measurement of shell formation process parameters for comparison with theoretical and modelling calculations;
- finding of optimal working parameters for specified shell size and quality.

REFERENCES

5 S.M.Tolokonnikov, V.S.Bushuev, A.I.Nikitenco, "The Particle Injector for the "Ballistic Furnace".", Report on this meeting.
Fig. 1. Schematic diagram of the "Ballistic furnace".
1 - optical registration unit; 2 - hot zone; 3 - fast preheating;
4 - Initial granules injector; 5 - cooling zone; 6 - fast forced cooling;
7 - electrical vacuum slide gate; 8 - formed shells collector;
9 - synchronization; 10 - temperature monitoring; 11 - pressure monitoring;
12 - injector control; 13 - fast cooling control; 14 - vacuum slide gate control;
15 - computer; 16 - heaters power supply; 17 - temperature control;
18 - gas supply; 19 - pressure control; 20 - vacuum equipment;
21 - cooling agent supply.
Fig. 2 Design of the "Ballistic furnace"

1 - hot zone; 2 - quartz tube; 3 - wire heater; 4 - thermoinsulator; 5 - electrical lead-in; 6 - window zone; 7 - initial granules feed; 8 - injector; 9 - angle adjustment equipment; 10 - injector zone; 11 - gas inlet/outlet; 12 - cooling zone; 13 - electrical vacuum slide gate; 14 - blown shells collector.
Fig. 3. The "Ballistic furnace" control system.

1 - control computer; 2 - CAMAC bus; 3 - generator; 4 - counters; 5 - control module; 6 - analog-digital converter; 7 - multiplexer; 8 - heaters control; 9 - gas control; 10 - vacuum control; 11 - status module; 12 - injector power supply; 13 - injector; 14 - input temperature and pressure data; 15 - heaters power supply; 16 - gas valves; 17 - vacuum valves; 18 - input status data.
We continued development of a technology to produce hollow shell laser fusion fuel capsules starting with a depolymerizable mandrel. In this technique we use (alphamethylstyrene) (PAMS) beads or shells as mandrels which are overcoated with plasma polymer. The PAMS mandrel is then thermally depolymerized to gas phase monomer which diffuses away through the permeable and more thermally stable plasma polymer coating, leaving a hollow shell. We have developed methods for controlling the size of the PAMS mandrel by either grinding to make smaller sizes or melt sintering to form larger mandrels. Sphericity and surface finish are improved by heating the PAMS mandrels in hot water using a surfactant to prevent aggregation. Problems with shell bursting due to too rapid an evolution of monomer were controlled through the heating rate and related studies of the depolymerization kinetics. Using this technique we have made mandrels from 200 µm to 4 mm diameter with sphericity better than 2 µm and local surface finish better than 10 nm RMS. We characterized sphericity through each step of the process and found that distortion occurs mainly during pyrolysis. To eliminate this distortion, we are investigating methods for achieving a uniform thermal and stress environment during pyrolysis.

*Work performed under the auspices of the U.S. Department of Energy Lawrence Livermore National Laboratory under contract number W-7405-ENG-48.
Fabrication of Hollow Shell ICF Targets Using a Deploymerizable Mandrel

S.A. Letts, E.M. Fearon, S.R. Buckley, M.D. Saculla, and R.C. Cook

Acknowledgements:
E.F. Lindsey, C.E. Moore, D. Schroen-Carey (WJSA), M. Whitman (UR/LLE)

Tenth Target Fabrication Specialists' Meeting
Taos, New Mexico
February 6 – 9, 1995
Objectives of the depolymerizing mandrel project

Overall objectives

- Preparation of a wide range of sizes of spherical shells (0.1 to 4 mm)
- Diagnostic layers at inside surface
- Texture at inside surface

Process goals

- Prepare spherical, smooth mandrels
- Deposit smooth, uniform coatings
- Control pyrolysis rate
- Measure shrinkage and sphericity

Status of the fabrication of shells by the depolymerizable mandrel technique

At previous TSM
- Formation of PAMS beads
- Surface finish
- Thermal analysis

Problems encountered
- Shrinkage
- Sphericity
- Collapse
- Bubble formation
- Residue

Current issues
- Molecular weight effects on bubble formation
- Mechanics of maintaining sphericity through Pyrolysis

- Diagnostic layer and texture on inner surface
- Shell mandrels from PAMS
A possible method for shell production uses thermally stable coating over a depolymerizable substrate.

CH₂

\( \Rightarrow CH₂ \quad (\text{CH₂ - C})_n \quad \text{Heat} \quad 280°C \quad \Rightarrow CH₂ = C \)

poly (α-methyl styrene) α-methyl styrene

**Polymer shell**

**Polymer shell with metal layer**

Smooth spherical PαMS mandrels are made using thermal and vapor treatment.
Thermal analysis shows that plasma polymer is stable at 300°C where PαMS decomposes to monomer.

Crushed PαMS beads are hot water treated to form spheres.
Plasma coating at high power results in bubble formation in the PAMS bead

- Bead softens at higher temperature (~60°C)
- Diameter swells—coating occasionally cracks
- Prevented by deposition at lower power for first ten hours

Most collapsed pyrolyzed shells were coated using the rolling pan technique which produces non-uniform coating.

Diameter = 2.3 mm

Optical

SEM

Diameter 620 µm

Diameter 780 µm
The heat of plasma polymer coating can cause bubbles to grow within PAMS beads

- Bubbles grow inside 11K Mw PAMS beads during plasma polymer coating
- Imbalanced beads (caused by bubbles) lead to nonuniform coating thickness
- Use of 19K Mw PAMS prevents bubble formation

SEM of completed pyrolyzed CH coated PAMS shows good sphericity and surface finish

Diameter = 650 μm  Wall Thickness = 33 μm
PAMS process development required consistent characterization

- Wall thickness calculated from M and D, compare with coater
- Shells with residue were distorted
- Shrinkage results:

<table>
<thead>
<tr>
<th>Pyrolysis time (minutes)</th>
<th>1000</th>
<th>2000</th>
<th>4000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pyrolysis</td>
<td>3</td>
<td>6</td>
<td>10</td>
</tr>
<tr>
<td>Temperature</td>
<td>5</td>
<td>10</td>
<td>15</td>
</tr>
</tbody>
</table>

The inner surface of the plasma polymer shell shows no roughness or particulate contamination

Diameter = 680μm  Wall thickness = 30 μm
The Sphere Mapper AFM characterizes sphericity and surface finish through the entire process.

Rotation during pyrolysis results in shells with better sphericity.

Surface tension forces from liquid PAMS during pyrolysis may cause distortion of the shell.
Pyrolysis with rotation results in improved sphericity

Shells up to 2 mm were made using a concentric needle droplet generator
Microencapsulated shells show very good sphericity with a small number of surface defects.

Sphericity is maintained through pyrolysis using mandrel made by microencapsulation.

Pyrolyzed at 300°C, 2000 min.
Conclusions: Shells from PAMS mandrels

Mandrels
- PAMS mandrels (0.4 to 4 mm dia.) were made by heating in water or microencapsulation
- Sphericity of better than 200 nm and surface finish of 3.5 nm can be achieved on mandrels
- Surface finish can be improved by drop tower solvent vapor exposure

Coating
- Plasma coating can soften PAMS mandrels
- Use of 19 k Mw PAMS eliminates bubble formation

Pyrolysis
- Collapse occurs if coating is nonuniform (bubbles, bouncing)
- Shells shrink isotropically 10% during pyrolysis (300 °C/2000 min.)
- Preliminary results show rotary pyrolysis maintains sphericity

Issues remaining in the depolymerizable mandrel process for ICF target application

- Maintain sphericity through pyrolysis on production scale (rotation or fluidized bed pyrolysis)
- Achieve routine good surface finish on mandrels
- Coating with a permeation barrier for hydrogen retention
- Incorporation of diagnostic layer or texture for target experiments
We will report on our work to produce a foam shell for two possible applications: 1) as a liquid-layered cryogenic target on Omega Upgrade, and 2) as a design for the National Ignition Facility. This target consists of a roughly 1 mm diameter (2 mm for NIF) and 100 mm thick spherical low-density foam shell surrounding a central void. The outside of the foam is overcoated with full density polymer which must be topologically smooth. The technology for manufacturing this style of foam shell involves microencapsulation techniques developed at Osaka Universities ILE.

We will present our progress in adapting the trimethacrylate formulation route to these shells developed by the ILE group for Omega and NIF scale targets. In addition we will report upon our progress in adapting a resorcinol/formaldehyde (R/F) low density foam formulation developed at LLNL to the microencapsulation process. Properties of the R/F shells produced will be presented as well as proposed pathways for overcoating these shells.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Progress in Development of Resorcinol/Formaldehyde Foam Shells

George Overturf
Robert Cook
Steve Letts
Steve Buckley
Mike McClellan, WJSA
Diana Schroen-Carey, WJSA

Tenth Target Specialist’s Meeting
Taos, New Mexico
February 6, 1995
The group at Osaka has developed a foam shell technology based on methacrylate chemistry.

- Current LLNL produced "Osaka" shells have 1-2 μm cell size and are thus opaque, preventing optical characterization of supported fluid or solid fuel layers.

![Graph showing transmittance of different materials vs. wedge thickness.]

- This has motivated our development of smaller cell-sized foam shells based on organic aerogel technology developed at LLNL over the past 10 years.

- The Osaka Group has recently reported two new formulations that they indicate lead to smaller cell size (see M. Takagi poster). We intend to stay as closely coupled to the Osaka work as possible.

Foam shells offer alternatives to simple solid and liquid layers.

- Foam provides a symmetric support of liquid D2 or DT before freezing, with a fine mesh template for solid nucleation, that may result in a smoother inner surface.

- The foam can symmetrically support 90-100% of a thick liquid layer leaving only a few microns to be symmetrized by thermal gradient or free-fall techniques.

- The foam shell must allow optical access to characterize the fill. A liquid layer must be symmetrized and characterized in the target chamber immediately before the shot. The optical properties of the shell necessary to allow this are currently being determined.
Foam shells are produced using a droplet generator. Shell diameter and wall thickness are controlled by fluid flow rates.

R/F gels have smaller cell size resulting in higher transparency.
The group at Osaka has developed a foam shell based on trimethacrylate chemistries.

- LLNL has duplicated the process
- We have found that the transparency through the shells produced at LLNL is insufficient to allow for optical characterization of fuel fill. The opacity is largely due to the 1-2 μm cell size.
- This search for a more transparent foam shell has motivated our move towards organic aerogel technology.
- Recently the Osaka group has reported a smaller cell sized methacrylate foam using redox initiation (M. Takagi)

### Resorcinol/Formaldehyde Reactions

In a basic carbonate solution, resorcinol forms the resocinolate anion which is more strongly ortho para directing than the neutral diphenol because a full negative charge is present.

\[
\text{[Resorcinol]} + \text{base} \rightleftharpoons \text{[Resocinolate]} \]

Nucleophilic attack by the phenolate anion on formaldehyde yields a hydroxymethyl derivative of resorcinol.

\[
\begin{align*}
\text{[Resorcinol]} & \quad H^+ + CH_2OH \quad H^+ \\
\text{[Hydroxymethyl Resorcinol]} & \quad \text{plus other resonant forms}
\end{align*}
\]

Loss of an OH group causes the hydroxymethyl derivative to form a quinone-like intermediate which subsequently reacts with a phenoxide or another hydroxymethyl resorcinol to form methylene and methylene ether bridges.
Schematic diagram of the RF gelation mechanism

Substituted resorcinol

RF "Clusters" 70-100Å diameter

RF Gel individual beads 70-100Å diameter

TEM of an RF foam shows the "string of beads" morphology. Particles are 150 Å in diameter
Must convert from known system of a Water/Oil/Water emulsion to an Oil/Water/Oil

Solvents must meet multiple conditions

- Inner phase must be density matched to the polymer phase
- The inner and polymer phases must be totally immiscible
- Solvent must not react with constituents in the polymer phase or the surfactant(s)
- Solvent must be a liquid in the process temperature range

Surfactant systems must be re-evaluated for each solvent/polymer phase combination

R/F shells have been produced.

- We have produced shells in the 1-2 mm diameter range with 25 to 100 μm walls. Our control over these parameters needs improvement.

- Sphericity looks good, concentricity needs work. Wall densification is a problem.

- Our understanding of the coupling between chemistry and the final shell quality is growing steadily.

- We are beginning to explore overcoating routes, starting with simple plasma polymer deposition coatings.
- Fractured R/F shell.
  - Shell wall in right thickness range.
  - Shell density ~100 mg/cc.
  - Inner and outer surfaces are smooth over long lengthscales.
  - Some evidence of particulate on inner surface.

Solvents strongly affect the density of the final foam shells

Experiments have been performed to compare the water solubility into several solvents versus the densities of the resulting foam.

Beads with a formulation density of 50mg cm$^{-3}$ were processed and characterized by interferometry and mass/volume measurements.

<table>
<thead>
<tr>
<th>foam density (mg/cc)</th>
<th>sol. aq. in, @T (%) wt</th>
<th>sol. dens. (g/cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dibutyl phthalate</td>
<td>140</td>
<td>0.4600 20C</td>
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<tr>
<td>1-methyl naphthalene</td>
<td>83</td>
<td>0.0377 20C</td>
</tr>
<tr>
<td>dodecane</td>
<td>57</td>
<td>0.0085 25C</td>
</tr>
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</table>

At 20C, 300ml of dibutyl phthalate can solvate 0.31g of water. Unfortunately 100 "preforms" only contain 0.024g of water!

Gelation time has a secondary effect on density. Shells gelled for 15 minutes had density of around 100 mg cm$^{-3}$ while at 30 minutes the density was around 230 mg cm$^{-3}$.

It appears that water loss occurs on the same time scale as gelation.
A novel technique for levitating a submillimeter-size ball has been developed. Unlike the conventional gas-dynamic levitation scheme which only employs a levitator placed under the target which provides the force needed to levitate the ball, the present system has a gas collector placed above the target which shapes the flow field around the ball to stably confine it along the axis of symmetry of the levitation fixture. Using this system submillimeter-size balls were levitated for several hours while they were continuously rotated in the levitation field. The system also showed excellent immunity to external disturbances such as nonuniform temperature field and air currents, and changes in the pressure of the levitation gas. Due to these features the present system may be combined with an appropriate coating scheme for the noncontact coating of ICF targets. The operational principle and results showing the capability and robustness of the levitation scheme will be presented. A coating system designed to facilitate noncontact coating of ICF targets will be evaluated in conjunction with the present levitation scheme.
Target capsules for Inertial Confinement Fusion (ICF) experiments made of beryllium or beryllium-containing materials are very attractive for the high energy implosions anticipated in the National Ignition Facility (NIF). The challenges in preparing beryllium capsules that will allow for diffusional filling of the hydrogenic fuel and that have an appropriate surface roughness are significant.

This paper discusses the feasibility of preparing beryllium rich organo-beryllium coatings using plasma enhanced chemical vapor deposition (PECVD) as a route to prepare advanced NIF capsules. PECVD is a more generalized form of the organic plasma polymerization process we currently use to make target ablators. There are a number of organometallic beryllium compounds known which would be suitable precursors for this process. We will demonstrate a concept for the safe handling and plasma CVD of these compounds.

Experiments on a surrogate chemical system using tetramethylgermanium as the organometallic precursor demonstrate that coatings with a high degree of germanium metal incorporation can be prepared. Methods to determine the germanium-to-carbon ratio will be described and the results used to suggest how one might run the current plasma polymerization device to prepare useful organo-beryllium coatings.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Feasibility of Organo-Beryllium Target Mandrels Using Organo-Germanium PECVD as a Surrogate

Raymond M. Brusasco
Robert Cook
Target Science and Technology

Thomas Dittrich
X-Division
University of California
Lawrence Livermore National Laboratory

Presented at:
Target Fabrication Specialist Meeting
Taos, NM
February 6-9, 1995
Plasma polymerization is a viable synthetic route for beryllium containing capsules.

- Organo-beryllium coatings are attractive for use as an ICF capsule material.
- The feasibility of preparing this material is indicated based on consideration of beryllium chemistry and extrapolations of a surrogate chemical system founded on organo-germanium coatings.
- The basic technology to prepare these coatings is in place.
- The safety issues are nontrivial but are very manageable.

A beryllium containing capsule must meet a rigorous set of performance criteria.

- Capable of fuel loading via diffusion filling
- Very smooth surface finish
- Tolerant of dopants as performance modifiers
- High deposition rate

Dopant levels and composition grading are high leverage items in NIF design.
Existing beryllium fabrication technologies may not be suitable for capsule formation.

**Machining**

Existing technology

Seams?

Full density Be (fuel fill)?

Surface finish?

**Plasma Spray**

Existing technology

High coating rates

Particle size (roughness)?

Thermal budget?

Air stability?

**Thermal Evaporation**

Existing technology

Roughness (columnar growth)?

We propose to prepare organo-beryllium coatings via plasma polymerization.

\[ \text{BeR}_2 \]

where R is:

- \(-\text{CH}_3\)
- \(-\text{C}_2\text{H}_5\)
- \(-\text{t-C(CH}_3)_3\)

Our expectation is a beryllium-carbon polymer with an amorphous structure, hence smooth surface characteristics.

- The organo-beryllium compounds are easily prepared (the diethyl compound is commercially available).

- Vapor pressures vary from 500 mTorr \((100^\circ C)\) for \(\text{Be(CH}_3)_2\) to 40 Torr \((25^\circ C)\) for \(\text{Be}(\text{C(CH}_3)_3)_2\)
A beryllium-carbon polymer is a credible capsule material.

- Main fuel kinetic energy is a figure of merit for ablator performance
- Increasing the carbon content increases the main fuel kinetic energy at burn through

Beryllium-carbon plasma polymer is assumed to be a highly metal doped CH-like material.

Plain Plasma Polymer

- Highly cross-linked polymer
- Amorphous phase
- Residual radicals
- Significant unsaturation

Be-C Polymer

- Heavy Be incorporation into polymer network
- Possible incorporation of beryllium hydride phase
  - Dispersed?
  - "Grains"?
Tetramethylgermane (TMG) is a reasonable surrogate for BeR$_2$.

- Safe, easy-to-handle metal alkyl
- Non-oxygen containing precursor
- In-house experience with TMG in plasma polymerization
- Literature studies indicate possible high metal-to-carbon ratios (7.4:1)* with either semiconductor or "organic-like" characteristics.**


A glass optical fiber makes a convenient, non-perturbing axial coating substrate.

- 125 μm diameter
- $\rho = 2.2$ g cm$^{-3}$
- Index modifier = Ge

- Break fiber into two segments - determine [Ge] by X-Ray Microfluorescence
- Characterize coating thickness by SEM
- Compute volume assuming right cylinders
- Determine mass of coating by weighing - subtract mass of glass based on volume and density
- Assume generic formula is $C_x$Ge$_y$, determine x and y

- H$_2$ flow rate 13 sccm
- TMG flow rate 0.27 sccm
- Chamber pressure 70 mTorr
- RF Power 12 W
- Coating time 4 hours
Coatings with significant germanium content are formed in the helical resonator coater.

The thinnest coatings did not survive handling for mass determination.
Within the plasma envelope, the coatings are heavily doped with germanium.

\[ C_x Ge_y \]

- No evidence for localized, enhanced upstream Ge concentration (c.f. Sadhir et al.)
- Considerable metal doping of plasma polymer is possible - extrapolation to beryllium is plausible.

Safety is a major concern for organo-beryllium precursors.

\[ BeR_2 \]

- Pyrophoric - burns spontaneously in air to BeO
- Toxic - Berylliosis

Exhaust Treatment

Glove Box

Ar in

Grignard Reaction Column

Solvents
A comprehensive system architecture provides safety and control of the coating process.

Plasma polymerization to produce organo-beryllium coatings is both feasible and manageable.

- Extrapolations from germanium system indicate that significant metal doping could be expected.
- Toxic nature of beryllium and its precursors must be carefully addressed but is manageable.
- Issue of surface roughness remains open.

Acknowledgments

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Ed Lindsey
Craig Moore
Bob Turner
Larry Witt

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
DESTRUCTIVE TESTER FOR THE ANALYSIS OF FILL PRESSURE AND COMPOSITION OF SINGLE ICF SHELLS

Roy Mangano and Richard Stephens
Fusion Group, General Atomics
P.O. Box 85608, San Diego, CA 92186-9784

The shell crusher system was designed for the destructive characterization of gas filled ICF shells. Shells to be tested range in diameters from 0.3 mm to 1.5 mm, with fill pressures ranging from 0.1 atm to 1.0 atm. The shell crusher consists of a Mitutoyo Digimatic depth micrometer modified to minimize system dead volume and provide for vacuum seal and ease of shell insertion. A minimum dead volume (=0.015 cc) pressure transducer was developed for measuring the target fill pressure (minimization of dead volume is required, since the shell volume is small and fill pressures are low). A gas chromatographic system was used to analyze gas composition. We will discuss the development of the micro-vacuum system, and present test results from sample shells.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
Inspiration from prev target specialists meeting

• Small Volume Tritium Fill System
  - M.A. Salazar, P.L. Gobby, H. Bush, J.K. Hoffer, and L.R. Foreman, LANL

• A destructive technique for measuring the fill of a fuel container
  - M.D. Saculla and S.A. Letts, LLNL

Needed for analysis of diagnostic gas fills in PVA drop tower

• Shell I.D. 0.3 to 2 mm
  — volume $10^{-3}$ µL

• Gas Pressure 0.1 to 1 atm
  — mass $1-10^4$ picomoles

• Gas Composition
  — diagnostic: Ar
  — potential contaminants: N$_2$, O$_2$, CO$_2$, H
Designed a small volume shell crusher as entry port to a Gas Chromatograph

Minimize volume of components

- capillary tubing
  - 7.7 μL connections
  - 10 μL injection loop
- high pressure valve
  - 1.6 μL
- micrometer spindle acts as crusher and calibrated volume
  - 10 μL in operation
  - 2 μL (min) - 80 μL (max)
- modify pressure gauge to minimize dead volume
  - volume 10 μL- reduced by 35 times

⇒ Total Volume 40 μL (shell volume 0.01 to 4 μL)
⇒ Minimum Pressure 0.03 Torr
Shell Crusher Schematic

Pressure Transducer Schematic

- Setra Model 239 Pressure Transducer
  - minimum pressure 0.01 Torr
  - separately pumped vacuum reference
- Head modified to reduce dead volume to 10 μL
Measurement Procedure

- Preload Mole Sieve Column with O₂
  — allows separation of O₂ and Ar

- Load & Inject calibration gas
  — 100 µL injection loop
  — 20 ppm Ar, O₂, N₂ in He

- Load shell & evacuate crusher volume
  — evacuate to 10⁻⁵ Torr

- Crush shell & determine pressure
  — determine volume by withdrawing spindle until pressure is halved
  — correct by ratio of shell volume to measurement volume

- Inject shell gas to selected column
  — 25% of shell gas

- Second injection possible
  — 20% of shell gas
Calibration gas injection similar to that from shells

Calibration; 1 cc sample

- $N_2$: 1.2 ppm
- $CO$: 2.1 ppm
- $CO_2$: 2.5 ppm
- $Ar$: 2.8 ppm
- $Ne$: 2.5 ppm
- $O_2$: 4.1 ppm
- $CH_4$: 2.0 ppm
- $H_2$: 2.7 ppm
- $He$: balance

Temperature:
- MS5A: 110°
- HSDB: 60°
- Detector: 110°

Flow rate:
- Detector A: 45 ml/min

Initial tests show sufficient sensitivity

2 Ar shells
- 0.1 atm, 0.5 mm dia

Vacuum blank
Summary

- Detect gas from individual shells
  - pressure transducer still to be assembled
- Analyze for a wide range of compositions
  - detect to 10% for low pressure & low volume shells
  - detect to 1% otherwise

⇒ Flexible tool for process development feed-back
Measuring capsule sphericity has become an integral part of LLNL’s characterization of ICF target capsules. We have designed and built a sphere mapping system incorporating an atomic force microscope and a precision air bearing that allows us to measure circumferential traces with ~0.5 μm lateral and sub-nanometer vertical resolution. We will describe the system and discuss some of the mechanical, electronic, and software problems that had to be addressed. We will also show some of the methods we have devised through use of the sphere mapper to ease the characterization process for the operator.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Motivation and Application

- Surface topography can strongly affect capsule performance
  - We have developed a Rotary Profilometer capable of characterizing spherical topography

- This device provides:
  - quality control for production
  - process feedback for new materials
  - data for "Bumpy Ball" experiments
Rotary Profilometer System Layout

- 486 PC with Digital Instruments Hardware and Software
- Mac Quadra with Data Acquisition Card
- Digital Instruments Scan Driver
- AFM Probe
- Air Bearing Spindle
- Rotary Encoder Interface
System Specifications

- **AFM**
  - subnanometer vertical resolution
  - 10nm probe tip radius
  - 10um probe tip height

- **Rotary Stage**
  - synchronous radial runout < 25nm
  - wobble < 0.1 uRadians

- **Total System Noise**
  - Vibration + Electrical < 5nm measured

- **Data Acquisition**
  - sampling rate 100 samples per 0.1° @ 1RPM
  - 3600 samples per rotation (averaged from 360000 @ 1RPM)
  - 16 bit ADC (0.6 Angstrom resolution)

- **Software**
  - Graphical User Interface (LabView)
  - Automatic sample centering calculation
  - Self calibrating RPM adjustment
  - All acquisition parameters defined by front panel controls

Two Views are Needed to Accurately Position the Shell on the Instrument

Side View as Seen from Camera

Top-Down View as Seen from Tool Scope
Comparasion of Precision Ball Bearing and Typical Mandrel

Winsted Precision Ball Bearing

Mandrel - 2SBT177#2

Rotary Profilometer Scan of Laser Ablated Capsule. "Soccer Ball" Pattern.
# ROTARY PROFILOMETER USER INTERFACE

<table>
<thead>
<tr>
<th>Multi Sphere Graph</th>
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Direct-drive inertial-fusion capsules must possess a high degree of spherical symmetry including concentricity of the inner and outer wall surfaces. When viewed with a compound microscope using transmitted narrow-bandwidth light, such as that produced with a thin-film interference filter, self-interference fringes appear within the image of a capsule that are localized to its equatorial plane. The fringes are distinct concentric rings when the capsule is uniform, whereas faint, distorted, or discontinuous rings indicate nonuniformity. We have formulated a model that predicts this self-interference pattern as a function of capsule parameters; it agrees well with actual measurements from the image of a well-characterized capsule. The wavefronts directly transmitted through the capsule and wavefronts that are multiple-reflected within its walls interfere to produce these rings. These wavefronts are constructed by tracing a single paraxial ray through the capsule and including reflections and third-order spherical aberration. The diameters of the rings are predicted from the intersection of the wavefronts. Changes in wall thickness as small as 10 nm affect the self-interference rings' diameters by several micrometers, whereas the ring diameters are independent of both the internal gas-fill pressure and the angle that rays impinge on the capsule. In addition, the diameters of the self-interference rings, when normalized to the capsule's outside diameter, are nearly independent of the outside diameter of the capsule.

This work was supported by the U.S. Department of Energy Office of Inertial Confinement Fusion under Cooperative Agreement No. DE-FC03-92SF19460, the University of Rochester, and the New York State Energy Research and Development Authority. The support of DOE does not constitute an endorsement by DOE of the views expressed in this article.
Determination of the Wall Thickness and Uniformity of Inertial-Fusion Capsules Using the Self-Interference Fringes Produced with Narrow-Bandwidth Illumination

M. D. Wittman, H. Kim, and A. S. Chow
University of Rochester
Laboratory for Laser Energetics

Tenth Target Fabrication Specialists' Meeting
Taos, New Mexico
6–10 February 1995
Summary
Self-interference patterns can be used to determine the wall thickness and uniformity of capsules.

- Self-interference fringes appear within the image of a capsule when viewed with a compound microscope using transmitted narrow-bandwidth light.
- The fringes are distinct concentric rings when the capsule is uniform, whereas faint, distorted, or discontinuous rings indicate nonuniformity.
- We have formulated a model that predicts this self-interference pattern (SIP) as a function of capsule parameters, and it agrees well with actual measurements from the image of a well-characterized capsule.
- This method is currently being used for preliminary selection of capsules from a batch of shells based on uniformity and approximate wall thickness.

A self-interference pattern (SIP) appears when a capsule is illuminated with narrow-bandwidth light.
Computer-generated SIP’s can be produced using a straightforward optical model

- Three sets of paraxial rays are traced through the capsule: a transmitted ray, a ray that is reflected twice in the first wall of the capsule, and another ray that is reflected twice in its second wall.
- The region of localization of the SIP fringes is determined from the plane of intersection of these rays.
- Third-order spherical aberration is included, and the cross-sectional profiles of the wavefronts are determined.
- The phase difference between the wavefronts is adjusted using their respective optical path differences, and the wavefronts are superposed at the plane of localization.
- The bright fringes in the SIP occur where the wavefronts intersect.
- The effects of wall-thickness nonuniformities along the axis of rotational symmetry of the capsule can also be determined.

The SIP results from interference between the transmitted and multiply-reflected wavefronts

![Ray diagram](image)
The intersections of the computer-generated wavefronts determine the SIP fringe locations

A computer-generated template is produced to indicate the bright-fringe locations in the SIP
The number of bright fringes in the SIP is used to determine capsule's approximate wall thickness.

The bright-fringe locations in the SIP can be used to indicate small differences in capsules' wall thickness.
The bright-fringe diameters in the SIP are strongly dependent on the capsule's wall thickness.

<table>
<thead>
<tr>
<th>Wall thickness (µm)</th>
<th>Calculated ring diameters (µm)</th>
<th>Measured ring diameters (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.89±0.05 µm (measured)</td>
<td>94 160 202 238</td>
<td>112±5 167±3 206±3</td>
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<tr>
<td>2.93</td>
<td>110 168 210 242</td>
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</table>

OD = 255±5 µm  
λ = 546 nm  
n = 1.4648±0.0003

The wall thickness of capsules can be determined from the bright-fringe diameters using look-up tables.

![Look-up table diagram](image-url)
A capsule's wall thickness can be determined uniquely by quantifying the SIP at more than one wavelength.

<table>
<thead>
<tr>
<th>Wavelength (nm)</th>
<th>Calculated ring diameters (μm)</th>
<th>Measured ring diameters (μm)</th>
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<tbody>
<tr>
<td>546 (n = 1.480)</td>
<td>64</td>
<td>67±5</td>
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<td>140</td>
<td>142±3</td>
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<td>184</td>
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<td>220</td>
<td>218±3</td>
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<tr>
<td>488 (n = 1.485)</td>
<td>60</td>
<td>63±5</td>
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<td>132</td>
<td>129±3</td>
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<td>176</td>
<td>173±3</td>
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<td>208</td>
<td>207±3</td>
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OD = 246±5 μm
WT = 3.00±0.05 μm

The normalized SIP fringe diameters are (nearly) independent of capsules' outside diameter.

OD = 250.0 μm
WT = 5.00 μm
n = 1.5000

OD = 500.0 μm
WT = 5.00 μm
n = 1.5000

OD = 1000.0 μm
WT = 5.00 μm
n = 1.5000
Irregular or low-contrast SIP’s indicate wall-thickness nonuniformities as small as a percent

**Conclusion**

The SIP has several interesting and useful properties

- The SIP is independent of the angle with which rays impinge on the capsule—a diffuse, narrow-bandwidth source can be used for illumination.

- The SIP is independent of the gas and fill pressure inside the capsule—the wall thickness of filled capsules can be determined.

- The normalized diameters of the bright fringes in the SIP are (nearly) independent of the capsules’ outside diameter (slight differences occur for thick-walled capsules)—only a single look-up table is required.

- The SIP is as sensitive to slight differences in wall thickness as dual-beam interferometry—only an inexpensive compound microscope equipped with a narrow-band filter is required to characterize capsules.
Characterization of foam shells by optical interferometric techniques is limited by scattering caused by the foam structure. Such scattering reduces the transmitted intensity as well as lowers the contrast of the fringes. Additionally, fringe position is not a sensitive detector of shell defects; because of the low density of the foam, shell non-concentricity or local defects cause only slight changes in the fringe position. We have used model calculations to estimate foam parameters required to allow adequate foam shell characterization. The acceptability of presently made foam will be discussed.
Characterization of Foam Shells

Richard B. Stephens
General Atomics

Tenth Target Fabrication Specialists’ Meeting
6-10 February 1995
Several Techniques are useful

- X-ray radiography
- Edge location measurement
- Interference Fringe Shift

⇒ All techniques have limitations


Limitations from Foam Structure

- Low mass density causes poor x-ray visibility
- Fine structure darkens image
- Large scale fluctuations reduce transparency
Foam edge contrast is low in X-radiography

- Simulated 1mm shell with 100 μm foam shows faint inner edge

[Image: Shell Cross section]

2nd radial derivative of Intensity vs radius.

[Graph: 2nd radial derivative of intensity vs radius]

---

**GENERAL ATOMICS**

Characterize fluctuations w/ x-radiography

- Image shows mass integral through shell with <1μm resolution

[Images: as taken, 5 μm average]

- 200 μm square images at the center of a foam sphere 540 μm dia
- 5 μm ≈ mode 300

- Measure fluctuations against curvature
  - regions = 25μm dia show fluct= 1% in mass -> density fluct
  =5%

⇒ Observe smaller scale w/ thinner samples
⇒ Analyze w/ FFT adjusted for nonlinearity

---

GENERAL ATOMICS
Fluctuation causes refraction

- Optical path length \( \propto \) index of refraction \( \propto \) density
  \[ L_{\text{opt}} = L \times n \]
- Angular deviation for fluctuation w/ wavelength \( \lambda \)
  \[ \sigma(\lambda) \propto \Delta n(\lambda) \]
- Deviation grows as sq rt of path length
  \[ \sigma(L,\lambda) \propto \Delta n(\lambda) \sqrt{\frac{L}{\lambda}} \]

GENERAL ATOMICS

Edge Images are blurred by ray misorientation

- ray uncertainty at exit
  - in angle
    \[ \sigma = \sqrt{\frac{L}{c}} \sigma \]
  - in offset
    \[ \sigma = \sqrt{Lc} \sigma \]

- blurs images behind foam
  \[ b = \sqrt{L^2 \sigma^2 + \sigma^2} \approx L \sigma \]
Density fluctuation ($\lambda \approx 1 \mu m$) < 1% for 1 $\mu m$ blur

- The allowed angular deviation reduced by wall thickness
  $$\sigma = \frac{b}{L} = 0.003$$
- The allowed angular deviation in 1 wavelength reduced further by sqrt of wall thickness
  $$\sigma(1 \mu m) = \sigma \sqrt{\frac{\Lambda}{L}} = 0.0002$$
- The allowed mass fluctuation % depends on foam density
  $$\Delta m/m = \Delta n/(n-1) = 0.006$$

Interference Fringes fade from path length dispersion

- Phase uncertainty in transmission
  - Dispersion in Path Length
  $$\Lambda = \sqrt{\frac{c}{L}} \ L \ S = \sqrt{\frac{cL}{2\pi}} \ \sigma$$

- Decreases coherence at focus
  - fringe contrast reduced
  $$f(\frac{1}{\Lambda}) = \exp\left(-\frac{\Lambda^2}{2\lambda^2}\right) = \exp\left(-\frac{cL}{4\pi\lambda^2} \ \sigma^2\right)$$
Fringe contrast reduction coupled to image blurring

\[ f(b) = e^{-\frac{c}{4\pi L}b^2} \]

where:
- \( b \) = blur width
- \( f \) = fringe contrast
- \( L \) = foam thickness
- \( c \) = cell size
- \( \lambda \) = wavelength

- Contrast = 99% required
  for 1 \( \mu \text{m} \) edge resolution
  (1 mm shell w/ 100 \( \mu \text{m} \) foam wall)

**General Atomics**

**Low foam density**

\( \Rightarrow \) slight fringe shift with non-concentricity

<table>
<thead>
<tr>
<th>Empty foam</th>
<th>EtOH filled Foam</th>
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<tr>
<td>offset = 2.6 ( \mu \text{m}/\mu \text{m} )</td>
<td>offset = 6.6( \mu \text{m}/\mu \text{m} )</td>
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</table>

(1 mm dia shell, 10 \( \mu \text{m} \) shell, 90 \( \mu \text{m} \) thick 5\% dense foam with 10 \( \mu \text{m} \) nonconc on inner surface)
Fringe Centers become harder to locate when contrast decreases

Radial Averaging can determine fringe center accurately

- Simulation shows edge offset from fringes
  (1 mm dia, 10 μm shell, 90 μm thick 5% dense foam)

- Reality is not so neat, but feasible
Characterizing foam shells will be difficult

- Low Density makes x-radiography hard
  - high resolution images help

- Edges or fringes need to be located to ≈ 1 μm
  - density fluctuations blur edge images
  - fringes are not sharply defined

- Radial averaging of images might help.
The peculiar properties of the photon tunneling process, which have been known since the time of Sir Issac Newton, are uniquely suited for use in characterizing the NIKE laser target films. We present here the scientific principles underlying the basic photon tunneling microscope and the commercial instrument which utilizes those principles. Details of the calibration techniques and some of the operational limitations of the instrument are presented. The vertical resolution of a planar surface by the PTM appears to be about one nanometer while the lateral resolution is about one micrometer. The nominal field of view of our instrument is a 100 micrometer diameter circle. Some comparisons are made of the PTM characteristics with those of other surface profiling instruments.

Characterization results of some of the polystyrene films used for NIKE targets are presented together with Fourier analyses of the film profiles.

Some possible extensions of the photon tunneling principles to characterization of solid cryogenic surfaces are also presented together with means by which the technique can be implemented.

Work supported by U.S. Department of Energy under Contract No.DE-AC03-91SF18601.
CHARACTERIZATION OF ALUMINUM PLATES AN FOAM FOR ICF 'CRACK' EXPERIMENTS

Norman E. Elliott and Peter L. Gobby
Los Alamos National Laboratory
P.O. Box 1663 MS-E549, Los Alamos, New Mexico 87545

Experiments to measure equation of state using Al plates and low density foam have been conducted for a number of years. Results were not always consistent and fabrication/assembly problems were a possible source of the inconsistencies. More careful characterization of the foam and Al plates has recently lead to improved experimental results. Various methods of characterization for the materials will be described as well as failures in measuring a real density of the materials.
CHARACTERIZATION OF ALUMINUM PLATES AND FOAM FOR ICF "CRACK" EXPERIMENTS

Norman Elliott
Peter Gobby
Vivian Gurule

Los Alamos National Laboratory
MST-7, POLYMERS & COATINGS
Los Alamos, New Mexico
“Crack” Experiment

- Foam captured between 2 Al plates
- Package held inside hohlraum
- Backlight x-ray generator allows measurement of crack closure during implosion

Foam X-section

50μm  30μm  750μm
Construction Methods

• Al plates
  pulsed oxygen, PVD Al + lapping
  Al foil
  Pulsed oxygen, PVD Al
  all followed by machining

• Foam
  Hauser jig borer
  Willemin-Macodel CNC machine

Characterization parameters
• real density of finished Al plates and foam

Radiography density measurement method
• measure areal density using x-ray radiography and calibrated stepwedges

• measure physical thickness by Dektak, Zeiss optical profilometer, Biorad image shearing microscope

• Calculate physical density
Al plate problems

- Initial PVD deposit too thick, thin by lapping not successful

- Al foil cut and stacked for end milling, too soft

- 2nd PVD deposit cut and milled successfully
Foam problems

- Radiography of initial parts showed much higher than expected density

- CNC machined parts showed some parts 2X expected density

- Difficulty in determining base foam thickness, scatter in results
Results

- Careful characterization has allowed successful conclusion of "crack" experiments

- As usual we have learned to be less trusting of ourselves

- Sensitivity of relatively crude radiography system has proven excellent
Successful Nova implosion experiments require precise knowledge of the DD or DT fuel content of the capsule at shot time. At room temperature, hydrogen typically permeates from ICF capsules coated with polyvinyl alcohol (PVA) at a rate such that half the fuel will be lost in 20 to 40 hours, depending on the uniformity, thickness, and integrity of the PVA gas-barrier. Although most Nova target shots occur within eight hours after the capsule is filled with fuel, shell-to-shell variability in hydrogen out-gassing rates means that occasionally an especially fast leaking shell will produce erroneous data. To address this variability, we have developed an efficient method to accurately predict the hydrogen retention properties of individual capsules. This method relies on the correlation of hydrogen permeation rates to argon permeation rates. Determination of the argon permeation rate, which is conveniently measured by X-ray fluorescence (XRF), allows the hydrogen permeation rate to be readily determined.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
USING X-RAY FLUORESCENCE TO RAPIDLY DETERMINE THE RATE AT WHICH HYDROGEN LEAKS FROM ICF CAPSULES

Tenth Target Fabrication Specialists' Meeting
February 6 - 10, 1995
Taos, New Mexico

Presented by: Dave Steinman
Martin Hoppe
IT IS ESSENTIAL TO KNOW THE RATE AT WHICH HYDROGEN ISOTOPES LEAK FROM ICF CAPSULES

- Analysis of implosion experiments requires precise knowledge of capsule fuel content

- Nova capsules typically leak half their fuel in 20 - 40 hours at room temperature

- Capsules may leak for several hours before they are imploded

THE RATE AT WHICH A CAPSULE LEAKS DETERMINES ITS "HALF-LIFE"

Definition

Capsule Half-Life: The time it takes for half the fill gas to leak from a capsule at a given temperature

- The half-life of a Nova capsule depends on the quality of the ~3 μm thick PVA gas barrier layer

- PVA thickness alone is not an accurate predictor of a capsule's half-life
OTHER CAPSULE HALF-LIFE MEASUREMENT TECHNIQUES HAVE SIGNIFICANT SHORT-COMINGS

Cryogenic and interferometric techniques....

- require ~50 atm capsule fills then monitoring fill pressure over tens of hours to determine leak rate
  - several days to fill capsules
  - capsules are stressed and may be damaged
- are labor intensive
- are not suited to mass production

OUR TECHNIQUE USES TWO CAPABILITIES RECENTLY BROUGHT ON LINE AT GA

These capabilities are:

- X-Ray Fluorescence (XRF) - to measure capsule argon content

- "High temperature" permeation filling

Note: On average, a ~3 μm PVA layer gives a capsule an argon half-life of 20 hours at 110° C
WE MADE A SIMPLE HYPOTHESIS:

The rate at which argon leaks from a capsule at 110° C directly correlates to the hydrogen leak rate at room temperature

IF : @ 110° C for 20 hours

THEN : @ 20° C for 30 hours

TO TEST OUR HYPOTHESIS WE FILLED CAPSULES FIRST WITH ARGON AND THEN WITH DEUTERIUM

1) Baked 10 capsules in a sealed container with 1 atm Ar at 110° C for 20 hours

2) Measured the Ar content of each capsule using XRF then calculated their Ar half-lives

3) Filled the capsules with 50 atm of DD

4) Measured the DD pressure in each capsule over 44 hours using interferometry to determine their DD half-lives

5) Calculated the correlation between Ar and DD half-lives
WE TOOK TWO SETS OF XRF MEASUREMENTS TO ENHANCE OUR QUALITY CONTROL

<table>
<thead>
<tr>
<th></th>
<th>After Fill</th>
<th>1st XRF</th>
<th>2nd XRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Good Shell</td>
<td></td>
<td><img src="image" alt="Filled" /></td>
<td><img src="image" alt="Filled" /></td>
</tr>
<tr>
<td>Leaky Shell</td>
<td><img src="image" alt="Leak" /></td>
<td><img src="image" alt="Leak" /></td>
<td><img src="image" alt="Leak" /></td>
</tr>
</tbody>
</table>

XRF DETERMINATION OF CAPSULE ARGON HALF-LIFE WAS FAST AND EASY

Capsules filled at 110° C

![Bar Chart]

- Reading 1
- Reading 2

Argon Half-Life (hours)

Capsule Number

GENERAL ATOMICS
WE DETERMINED THE ROOM TEMPERATURE DD HALF-LIFE OF EACH CAPSULE

Interferometric Determination of Capsule Half-Life

This capsule's DD half-life was 22 hours

Fill Pressure (atm)

Time (hours)

INTERFEROMETRIC DETERMINATION OF CAPSULE DEUTERIUM HALF-LIFE WAS IMPRECISE

Capsules tested at 20° C

Deuterium Half-Life (hours)

Capsule Number
INTERFEROMETRIC MEASUREMENT OF EACH CAPSULE WAS LABORIOUS

- Two days to fill shells
- Several days monitoring leak rate
- Ambiguous fringe identification

We developed an eggcrate that permitted in-situ interferometric capsule measurement

Plug: See Martin Hoppe's poster

WE NORMALIZED THE DATA TO COMPARE ARGON TO DEUTERIUM CAPSULE HALF-LIVES
WE INTEND TO REFINE OUR MEASUREMENTS TO BETTER PREDICT CAPSULE HYDROGEN HALF-LIVES

Conclusion:

The rate at which hydrogen leaks from a capsule at room temperature can be predicted from the argon leak rate at elevated temperatures.

@ 110° C for 20 hours

@ 20° C for 30 hours

GENERAL ATOMICS
Precise information regarding dopant concentrations in ICF capsules are required for shot diagnostics. A simple but accurate method has been utilized to calibrate the Omicron XRF which does not rely on accurately knowing the theoretical or instrumental parameters. Argon and chlorine calibration was achieved using capsules containing known quantities of the element. XRF calibration for titanium and germanium was achieved by correlating the analyte signal of interest to the chlorine count ratio obtained in a thin film of material with a precisely known analyte/chlorine concentration. In addition, calibration of the argon or germanium signal for capsules with wall thicknesses ranging to over 60 μm was accomplished empirically.
XRF CALIBRATION FOR ICF CAPSULE MEASUREMENTS ACHIEVED USING GA MANUFACTURED STANDARDS

Tenth Target Fabrication Specialists' Meeting

February 6 - 10, 1995

Taos, New Mexico

Presented by: Martin Hoppe
OMICRON XRF CALIBRATION ACHIEVED USING GA MANUFACTURED STANDARDS

• Advantages of Using Standards
  - Do not require precise information regarding instrumental geometries
  - Do not need to know precise information regarding x-ray energy spectrum, x-ray filter thickness, etc.
  - Improved accuracy
  - Simple, does not require detailed theoretical modeling

• Disadvantages
  - Requires the availability of suitable standards
  - Large changes in sample sizes or geometries may require recalibration

GA's OMICRON XRF IS CALIBRATED FOR ALL CURRENT DOPANTS IN STANDARD SIZE CAPSULES

• Argon
  - Ar in GDP/PVA/PS capsules to >60μm total wall

• Chlorine
  - Cl in bare mandrels (<5μm thick) quantitative
  - Cl in finished capsules qualitative

• Titanium
  - Ti in bare mandrels (<10μm thick) quantitative
  - Ti in finished capsules qualitative

• Germanium in GDP
  - Ge doping levels to ≥2.5 atomic %
  - Total wall thickness to ≥60μm
THE ABILITY TO MANUFACTURE SUITABLE STANDARDS WAS THE KEY

- **Argon Calibration**
  - Initial calibration achieved using stalk mounted shell provided courtesy of LLNL
  - Subsequent development of permeation filling of capsules to equilibrium with Ar after the coating steps yielded many suitable Ar standards
  - Permeation filled capsules readily allowed for Ar calibration as a function of total wall thickness

DETERMINATION OF THE XRF SENSITIVITY FOR CHLORINE IS FUNDAMENTAL FOR THE CALIBRATION OF OTHER ELEMENTS

- **Chlorine Calibration**
  - Determined XRF sensitivity for Cl using mandrels prepared from 0.97 Atom% Cl polychlorostyrene as analyzed in duplicate by the combustion method

<table>
<thead>
<tr>
<th>Shell #</th>
<th>OD(µm)</th>
<th>Wall(µm)</th>
<th>Cts/(Sec·Atom) x 10^{14}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>448</td>
<td>2.7</td>
<td>6.38</td>
</tr>
<tr>
<td>2</td>
<td>444</td>
<td>2.7</td>
<td>6.07</td>
</tr>
<tr>
<td>3</td>
<td>451</td>
<td>3.8</td>
<td>6.30</td>
</tr>
<tr>
<td>20</td>
<td>442</td>
<td>3.2</td>
<td>6.03</td>
</tr>
</tbody>
</table>

Average = 6.17±.15
Most Other Elements Can Be Calibrated by Referencing to Chlorine

- Most Elements in the Periodic Table Form Stable Chlorides
  - Pure compounds have a well defined analyte to chlorine ratio
  - By referencing the analyte signal to the chlorine signal, the total mass nor concentration of the sample in the x-ray beam does not need to be known

- Titanium and Germanium
  - Prepared very thin films of samples from compounds of known stoichiometry [(CsH5)2TiCl2; (CsH5)2GeCl2]
  - (CsH5)2TiCl2 was dissolved in a benzene/PS solution and cast as a thin film
  - (C6H5)2GeCl2 was analyzed as a neat film (<1 μm thick)

XRF Calibration for Germanium Was the Most Demanding

- At Desired Doping Levels Ge Substantially Attenuates Both the Exciting and Emitted X-rays
- Ge Counts vs Wall Thickness is Non-linear and Varies with Concentration
- The Molecular Matrix of GDP changes with Increased Ge Doping Levels
- Thus Corrections Were Required For
  - X-ray attenuation of the exciting and emitted x-rays
  - Changes in attenuation as a function of capsule wall thickness
  - Changes in attenuation as a function of Ge concentration
  - Changes in CH concentration as a function of Ge concentration
XRF CALIBRATION FOR GERMANIUM CONCENTRATION AND WALL THICKNESS IS DERIVED FROM $I = I_0 e^{-\frac{(\mu/\rho)t}{p}}$

- $I = I_0 e^{-\frac{(\mu/\rho)t}{p}} \Rightarrow \ln(I) = \ln(I_0) - \left(\frac{\mu}{\rho}\right)pt$  ($y=mx+b$)
  - $\mu/\rho$ is the "Effective" Mass Absorp. Coef.; $\rho$ is the Density; $t$ is Thickness
  - $I_0$ is set to be the true Ge concentration; $I$ is the observed Ge concentration.
  - The Only Parameter That Changes With Changing Ge Concentrations is $\rho$.

- The value for $(\mu/\rho)p$ was determined for GDP at 1.72At% Ge
  - Other concentrations are then determined iteratively until the value measured for $I$ is consistent with the value determined for $I_0$.

INITIAL CORRECTION DETERMINED FROM A SERIES OF SHELLS WITH IDENTICAL Ge CONCENTRATION BUT OF VARYING WALL THICKNESS

Uncorrected Ln(At% Ge) Vs GDP Wall Thickness ($\mu$m)

$slope = -\frac{(\mu/\rho)p}{[Ge]} = 1.72At\%$

At% Ge$_{obs} = 100x(C/S)$_$_{obs}/(C/SA)/Total \# of atoms in GDP
  - Assumes 1.3/1 ratio of H/C in GDP.
A CHANGE IN [Ge] RESULTS IN A CORRESPONDING CHANGE IN SLOPE - $(\mu/\rho)\rho$

Uncorrected $\ln(\text{At}\%\text{Ge})$ Vs GDP Wall Thickness ($\mu$m)

\[
\text{slope} = -(\mu/\rho)\rho
\]

$\ln(\text{At}\%\text{Ge}_{\text{obs}})$

GDP Wall Thickness ($\mu$m)

A CORRECTION FOR VOLUMETRIC EXPANSION COMPLETES THE Ge CALIBRATION

Expected Density of Ge-doped GDP as a Function of [Ge]

Data courtesy of LLNL
AFTER APPLYING CORRECTIONS SCATTER IN XRF RESULTS IS ACCEPTABLE

Corrected At% Ge Vs GDP Thickness

Average At%Ge is 1.72 with a Standard Deviation of 0.04

GENERAL ATOMICS
EMISSIVITY AND ACCOMMODATION COEFFICIENT OF POLYMER TARGET SHELL AT TEMPERATURES BETWEEN 200K AND 300K*

A. Honig, Q. Fan, C.-K. Hsu, and X. Wei
Physics Dept., Syracuse University
Syracuse, NY 13244

The emissivities and accommodation coefficients for helium and deuterium gas were measured for polystyrene target shells from several production batches. The shells varied in wall thickness, diameter and surface conditions as viewed with an optical microscope. The measuring technique was described at the previous Target Fabrication Specialists' Meeting held in Monterey¹. A shell is positioned in a small cavity, filled with a gas (helium or deuterium in this report) to a pressure of about 12 atmospheres, and then cooled to 77K, where the permeation rate is negligible. After evacuating the fill gas, the cavity is immersed in a thermostatic liquid at temperature $T_c$, selected within the range 250K to 350K, and the leak-up rate in the cavity is monitored with a Baratron. The key to the method is that the shell temperature, $T_s$, and its rate of change, $dT_s/dt$, can be determined from the derivative of the shell leak-out rate in conjunction with a prior shell permeation rate calibration. From these measured quantities and the known heat capacity of the shell, the heat absorbed by the shell, $dQ_s/dt$ is obtained. The emissivity and accommodation coefficient follow from an analysis in terms of $T_s$, $T_c$, and the pressure.

In the previous report¹, the shell was not sufficiently conductively isolated from the cavity. In the present experiments, support of the target shell by spider-silks across the fork of a target stalk does provide sufficient isolation. Emissivity and accommodation coefficient results will be presented. We observe large differences among the accommodation coefficients of shells whose surfaces appear indistinguishable under optical microscopy. This is not surprising since the accommodation coefficient depends on surface roughness on an atomic scale on nm. Post emissivity-measurement SEM scans are in progress, to correlate with our other measurements.

*Work supported by DOE under Grant No: DE-FG03-93SF20144
EMISSIVITY AND ACCOMMODATION COEFFICIENT OF POLYMER TARGET SHELLS AT TEMPERATURES BETWEEN 200K AND 300K*

A. Honig, Q. Fan, C.-K. Hsu and X. Wei

*Physics Department, Syracuse University, Syracuse, NY 13244

10th Target Fabrication Specialists' Meeting
Feb. 6-9, 1995, Taos, NM

Shells Provided by:

Roger Q. Gram, Steve Noyes and Hyogun Kim, LLE
Bob Cook, LLNL
Wayne Miller, General Atomics

*Work supported by DOE through NLUF Grant Number: DE-FG03-93SF20144.

Attendance at this meeting supported by General Atomics.
EMISSIVITY MEASUREMENT APPARATUS

EMISSIVITY MEASUREMENT PROCEDURE

1. With sample cell near room temperature (293K), permeate gas into target shell to about 12 atm pressure.

2. Immerse sample cell into liquid nitrogen (77K) bath.

3. Evacuate sample cell. At 77K, target shell inside sample cell is impermeable.

4. Remove liquid nitrogen bath at t = 0. At t = 1 min, immerse sample cell in large methanol thermal bath at selected temperature to fix the wall temperature of sample cell, Te.

5. Monitor P due to permeation of gas out of target shell as its temperature, Ts, rises due to heat transport from warmer sample cell walls. The heat is via radiation, and via free molecular gas conduction. The former depends on emissivity, and the latter depends on accommodation coefficient.

DETAIL OF SAMPLE CELL

THIS IS THE CONFIGURATION USED IN THE EXPERIMENTS REPORTED HERE.

HEAT CONDUCTION IS LOW WITH THIS TARGET MOUNT. TARGET SHELL IS GLUED TO SPIDER SILKS. MASS OF GLUE IS 3 μg.
DETAIL OF SAMPLE CELL WITH OPTICAL ACCESS

OPTICAL ACCESS IS USEFUL FOR CONTACTLESS THERMOMETRY AND FOR VISUAL OBSERVATION OF SHELL FRACTURE.

TARGET IS POSITIONED ON SPIDER-SILK NET WITHOUT GLUE.

HEAT TRANSFER MECHANISMS

RADIATION

\[ Q_s (RAD) = e_s \sigma A_s (T_{ref}^4 - T_s^4) \]

- \( e_s \) = emissivity of target shell
- \( \sigma = 5.67 \times 10^{-12} \) W/cm\(^2\)K\(^4\)
- \( A_s = \) area of target shell

GAS CONDUCTION IN FREE MOLECULAR CONDUCTION REGIME (\( P < 0.1 \) Pa)

\[ Q_s (RarefiedGas) = \alpha_s \Lambda (T_{ref} - T_s) \]

- \( \Lambda = \Lambda_0 \left( \frac{273}{T_s} \right) \)

- \( \alpha_s = \) accommodation coefficient
  (fraction of maximum transferable "hot" molecule's energy imparted to cold target shell upon collision)

Trick is to measure \( T_s \) without conducting contacts, and to separate out the two heat transport contributions.
SHELL TEMPERATURE ($T_s$) MEASUREMENT

SHELL OUTGASES WITH TIME CONSTANT $\tau$

$$\tau = \frac{WR}{3K_pRT_s}$$

$W$ = Wall Thickness,
$R$ = Shell Radius,
$K_p$ = Permeability Coefficient.

$K_p(T)$ has strong temperature dependence and is determined from semilog plot of $\tau T$ vs $T^{-1}$.

During a target shell warm-up, the temperature $T_s$ rises from heat transfer from radiation and gas conduction from the sample cell walls at $T_w$. Using the calibration of $K_p(T)$, $T_s$ is found from the instantaneous outgas rates $\tau(t)$, which are determined from the formula

$$\tau(t) = \frac{P_{final} - P(t)}{dP(t)/dt}.$$

(See A. Honig et al, 9th Target Fabrication Specialists' Meeting, July 6-8, 1993, Monterey, CA).

OTHER $T_s$ DETERMINATION METHODS:

Visual: phase transition
-size of the shell
-interference
ESR Curie law for shells.
-GDP coated fresh shells.
-Shells doped with paramagnetic material.

PERMEATION CALIBRATION OF SHELL #1S FOR He AND D$_2$ GAS

![Graph showing permeation calibration of Shell #1S for He and D$_2$ gas]

SHELL #1S: Polystyrene (microencapsulation method)
OD = 945 $\mu$m, MASS = 50.1 $\mu$g, WALL THICKNESS = 17 $\mu$m
PRESSURE EVOLUTION DUE TO OUTGASING OF TARGET SHELL #1S DURING WARM-UP. SHELL INITIALLY FILLED TO 12 ATM. HELIUM.

\[ P' = P - \text{background pressure (from sample cell wall outgasing)} \]

TEMPERATURE EVOLUTION OF TARGET SHELL #1S DURING WARM-UP. \( T_s \) CALCULATED FROM \( \frac{dP'}{dt} \) OF PREVIOUS FIGURE.
DETERMINATION OF EMISSIVITY AND ACCOMMODATION COEFFICIENT FOR SHELL #1S AT $T_w = 252K$.

SHELL #1S: Polystyrene, OD=945µm, MASS=50.1µg, WALL THICKNESS=17µm

Experimental $dQ/dt$ is $C_v(T) \times dT_s/dt$, where $C_v$ is heat capacity of shell.

$\alpha$ is adjusted to produce straight-line radiative heat transport curve, $e$.

$\alpha = 0.0037$ (with $P$ corrected for transpiration).

Emissivity = (slope of curve e)/$a=0.016$.

---

DETERMINATION OF EMISSIVITY AND ACCOMMODATION COEFFICIENT FOR SHELL #1S AT $T_w = 341K$.

SHELL #1S: Polystyrene, OD=945µm, MASS=50.1µg, WALL THICKNESS=17µm

Experimental $dQ/dt$ is $C_v(T) \times dT_s/dt$, where $C_v$ is heat capacity of shell.

$\alpha$ is adjusted to produce straight-line radiative heat transport curve, $e$.

$\alpha = 0.0027$ (with $P$ corrected for transpiration).

Emissivity = (slope of curve e)/$a=0.012$. 

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Emissivity(e) and Accommodation Coefficient(a) Determinations

**Summary**

1. Emissivities of polymer shells have been measured to about 10% accuracy in the temperature range 250 - 350K. The low values obtained for polystyrene shells, $\sim 10^{-2}$, allow slower shroud withdrawal rates for stable temperature cryogenic targets than were calculated previously using higher assumed emissivities.

2. Accommodation coefficients are obtained to about 10% accuracy in the 250 - 350 K temperature region. The low values, $\sim 3 \times 10^{-3}$, also imply slow warming of cryogenic target shells when significant residual gas is present in the fusion chamber.

3. The small accommodation coefficient values imply very smooth (on an atomic scale) polystyrene shell surfaces. There do not appear to be appreciable differences for He and D$_2$ gas. Among different shells, however, variation of accommodation coefficient is observed. We have not yet made extensive enough comparisons among SEM's and measured accommodation coefficients to establish an expected correlation.

4. A few shells behave anomalously, and cannot be fitted easily to a $T^4$ radiative warming dependence. This is believed due to pre-rupture effects*, mounts which are too conductive relative to the mass of the shells, and possibly occasional impurities in the fill gases.

5. The accuracy of all of these measurements can be improved using cleaner sample-cell walls.

6. Other contactless means of measuring shell temperature allow extension of this type measurement down to liquid hydrogen or liquid helium temperatures. We have already shown that magnetic susceptibility measured by electron spin resonance (ESR) on fresh GDP shells, or on polymer doped with paramagnetic material, can provide adequate thermometry. For optical methods of shell temperature determination, and visible observation of rupture phenomena, a sample cell with optical access has also been constructed.

Both Los Alamos and Lawrence Livermore Laboratories have studied capsule and laser driven target designs for the National Ignition Facility. A current hohlraum design is a 2.76 mm radius, 9.5 mm long gold cylinder with 1.39 mm radius laser entrance holes covered by 1 μm thick plastic foils. Laser beams strike the inside cylinder wall from two separate cones with a peak power less than 400 TW. The problem with a pressure pulse caused by wall plasma stagnating on axis has been overcome by filling the hohlraum with gas. Currently this is aqua-molar hydrogen-helium gas at 0.83 mg/cc density. One capsule uses a 160 μm plastic ablator doped with oxygen and bromine surrounding an 80 μm thick DT ice layer with an inner radius of 0.87 mm. Calculation of the combined hohlraum and capsule using 1.34 MJ of laser energy achieves a yield of 11.4 MJ.

The choice and composition of the ablator are sensitive to the peak radiation temperature driving the capsule implosion. Above about 300eV plastic ablators are viable. At 250eV and below, beryllium appears the best choice. Each ablator contains some high Z material as a dopant. As the peak driving temperature increases, so does the required dopant level. Beryllium driven at about 200eV tolerates no dopant. The choice of dopant is not critical, almost any high Z material will do. Homogeneity of the ablator is, however, critical. Bromine is often added to plastic because it can bond at the molecular level. Copper is suitable for beryllium since forms an alloy at appropriate concentrations. A beryllium capsule with a 155 μm thick ablator doped with 0.9 atom% copper, and the same inner dimensions as the plastic capsule, placed in a similar hohlraum, yields 6.9 MJ in a non-optimized calculation.

One dimensional calculations of the beryllium and plastic designs yield 17.4 and 14.5 MJ. Performance degradation in two dimensional calculations results from imperfect drive symmetry. By adjusting the relative power and pointing of two laser beam cones, the plastic capsule achieves nearly the one dimensional performance. The beryllium capsule yield can be similarly improved.

Surface roughness is another source of asymmetry. Two dimensional calculations of non-linear growth of outer surface finish perturbations on both capsules show a cliff in performance at about 50 nm roughness. Perturbations on the DT ice surface produce a performance cliff at about 4 μm in the beryllium capsule and 2 μm in the plastic. Both roughnesses are comparable to those measured on DT ice shells formed by the beta layering process.
HYDRODYNAMIC STABILITY OF BERYLLIUM CAPSULES

by

Douglas C. Wilson, Nelson M. Hoffman, and William J. Krauser

Los Alamos National Laboratory

Tenth Target Fabrication Specialists’ Meeting

February 7, 1995
The beryllium and plastic capsules are very similar.

Yield 17 MJ
Absorbed Energy ~200kJ
Convergence Ratio 28
Peak Areal Density (rR) ~1.15 g/cm²
Peak Fuel Density ~ 1800 g/cm³

Yield 15 MJ
Absorbed Energy ~150kJ
Convergence Ratio 36
Peak Areal Density (rR) ~1.3 g/cm²
Peak Fuel Density ~ 1600 g/cm³

Los Alamos

2D multimode calculations suggest both the beryllium and plastic capsules fail for ablator RMS surface roughness of about 50 nm

Twelve modes 4,8,12,...48 90-degree sector
With a 20 nm roughness, the beryllium capsule tolerates a 2% lower final temperature before failing.

2D multimode calculations suggest the beryllium capsule has comparable or less sensitivity to DT ice roughness than the plastic capsule.

Twelve modes 4, 8, 12, ..., 48 90-degree sector
Conclusions

• Beryllium capsules offer an alternative to plastic for indirect drive.
  Strong shells may be able to contain DT at room temperature.

• Copper, or any high Z atom, can be added to adjust the opacity.

• Perturbation growth is largest at wavelengths ($\lambda$) of 200-300 $\mu$m. The challenge is more to make a spherical, 2000$\mu$m diameter capsule, than a smooth ($\lambda < 50$ $\mu$m) one.

• Beryllium and plastic ablator designs have similar sensitivities to surface perturbations. At nominal drive ablator surface finish tolerances are about 50 nm and DT ice surfaces of 2000nm (2$\mu$m).

• At less than nominal final drive, sensitivity to outer surface finish is greatly enhanced. A 20nm surface allows 2% lower drive temperature, 7% lower requires more than 50 times smoother finish.

• Much work remains to understand the effects of joint imperfections and of three dimensional perturbations.

Los Alamos
We report the first measurements of the beta-layering process for D-T mixtures in a cell with a toroidal cross-section. This shape closely approximates the curvature of a sphere, but has the optical access of a cylinder. The widows of this cell can be heated slightly to remove the frozen hydrogen from the cell end windows. This feature allows high resolution optical probing of the layer itself without the worry that a defect in the DT ice covering the window will be interpreted as an asymmetry in the ice on the walls. We will present surface roughness data and give an estimate of the ultimate surface smoothness achievable by this process.
Preliminary Experiments in a Toroidal Cell using $D_2$

Jim Hoffer - Los Alamos
John Simpson - General Atomics, Inc.

The 10th Target Fabrication Specialists' Meeting

Taos, New Mexico, February 7, 1995

A toroidal cylinder is equivalent to an 'everted' sphere!

Cryogenic Targets for ICF
Solid Layering Techniques

- The Beta-Layering Phenomenon

The redistribution of solid D-T into a uniform, symmetric mass driven by radioactive self-heating.

- Start by freezing the fuel into an irregular mass
- Driven by beta decay, solid D-T is self-heated
- A parabolic temperature profile is established
- Thus, the outside of the solid is warmer than the interior
- Thick layers become warmer than thinner ones
- D-T evaporates from the surface of thicker layers
- The layer temperature improves tenfold every 61 min.
- Uniformity is achieved only in an isothermal shell

Beta-layering was not seen in small GNB's due to the thin layers and long time scales. It was first observed in 1987 at Los Alamos by Hoffer & Foreman.

LANL 75μm and 125μm DT Layers

- Cryogenic Targets for DT

125μm D-T Solid Layer & Empty Cell Power Spectra

LANL 75μm and 125μm DT Layers

191 μm D-T Layer Accumulative Surface Roughness

- LANL
- Beta
An 'verted' sphere at Omega-UPGRADE size

Cylindrical geometry permits high-resolution optics

Diagram of an empty toroidal cell

Double Ended Heating of D-T Cell Shows D₂ Layering

D₂ Layering Sequence at 15 K with 8 ma Window Heater Current
Maximum Entropy Deconvolution is a technique used to bring out fine detail in astronomical images by removing the optical spreading of a point source of light (a star) by the earth's atmosphere and the telescope optical train. This spreading function, or point spread function (psf), is determined by the optical quality of the medium through which light must propagate before being recorded or imaged. The spreading function is mixed, or convolved, with the optical information of interest, causing image smearing or blurring. The image blurring can be removed from the recorded image by a deconvolution process, creating an image with finer detail and a more precise rendition of the actual object of interest. This technique is shown to be applicable to the CCD images of beta layered D-T solid by imaging an artificial point source from which the point spread function can then be measured. The design of this artificial point source will be discussed, as well as the technique and procedures necessary to make an accurate measurement of the spreading, as light propagates through the optical train. Results of the point spread function measurements are presented, and the maximum entropy deconvolution of D-T solid layer images are demonstrated.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
Point Spread Function Measurements and Maximum Entropy Deconvolution

- In all optical systems, light rays from the object of interest are spread due to effects of the imaging system and the surrounding medium - resulting in image blurring or "convolution"

- Diffraction, optical aberrations, and the nature and dynamics of the enclosing medium (atmospheric clarity & turbulence or system vibrations) can all contribute to point spreading

- The spreading function, called point spread function (psf), can be characterized and utilized to remove the effects of convolution by performing an image analysis technique called Maximum Entropy Deconvolution

- Maximum Entropy Deconvolution is utilized to improve astronomical image quality for both optical (HST) and radio (VLA) astronomy

- Point Source image for optical configuration of interest must be available

Experimental Procedures and Observations

- Back illuminated pinhole having a diameter less than one pixel at the CCD image plane will behave as a point source

- For these experiments, one pixel subtends a length of 2.2 μm (focal reducer in) and 1.5 μm (focal reducer out) at the source/object plane

- Pinholes are placed at D-T cell location inside the cryostat cold can and imaged through cryostat windows and 2.5" Maksutov-Cassegrain catadioptric microscope optics

- Point Spread Function measurements are made with several pinhole sizes and optical configurations

- Optical aberrations are observed and verified through image defocusing and off-axis aperturing

- "Hidden Image" Maximum Entropy Deconvolution software package was purchased from Sehgal Corporation and is running on a ™Pentium 90 mhz processor
Backlighted Pinhole Images Show Point Source Cutoff

Point Source Image Spreading Causes Shift in Mapping of Pinhole to PSF Image Spot.
Intensity Distribution Curves For 0.5 μm - 8.0 μm Pinhole Images

Image Defocussing Shows Evidence of Astigmatism and Coma
1.0 μm Spot Defocussing May Show Evidence of Spherical Aberration

Spot Diagrams for Maksutov-Cassegrain Optics

Reprinted from Telescope Optics, H. Ruttun and M. van Veenooij
Summary

- Point Spread Function measurements made for several optical configurations and pinhole sizes
- For point source pinholes (0.5 - 3.0 \( \mu \text{m} \)), the optical image spreading was measured to be about 10 \( \mu \text{m} \) (fwhm)
- Optical aberrations observed included astigmatism, coma, and possibly spherical aberration
- Next step will be to utilize the acquired psf images to perform a maximum entropy deconvolution analysis on images of layered D-T solid surfaces to improve image quality and surface roughness measurement data
The current stage of the research in the field of Inertial Confinement Fusion (ICF) is characterized by going to much more powerful laser drivers (30 kJ - 2 MJ) and accordingly to using cryogenic targets with thick hydrogen fuel layers (10-180 μm). The typical target for these experiments consists of a hollow polymer capsule filled with a fuel at pressures of 70-1543 atm at 300K, which corresponds to gas-fuel filling density of 11-128 mg/ccm. We have studied experimentally the properties of deuterium, hydrogen and their 1:1 mixture enclosed inside glass microballoons (200-600 μm in diameter) in the temperature range of 4.2 to 300K, pressure range of 60 to 1200 atm and density range of 10 to 108 mg/ccm. The greatest cryolayer thickness inspected was about 54 μm for a liquid layer and about 25 μm for a solid layer.

Our data have shown that fast refreeze technique doesn’t provide a uniform solid layer inside the capsule if the average thickness of the cryolayer exceeds 20 μm. It has been found that dynamic of formation of liquid layers depends more on the gas filling density with respect to its critical value than on the value of liquid layer thickness.

The requirement that the layering module should be placed outside of the ICF target chamber calls for the creation of cryotargets with characteristics of the layer quality (smooth surface, thickness uniformity) which are stable for the significant length of time required for the transport of the target to the target chamber. Our investigations have shown that cryolayers of quasi-amorphous and single-crystal structures can conserve their quality characteristics for a comparatively long period (more than 45 min) if maintained in cryogenic, isothermal conditions.

The unique methods used to make these targets, called the Fall and Strike Technique (FST) and the Impact Load Technique (ILT), which allow one to create these cryolayer structures inside the actual ICF target, have been developed by the Cryotarget Group at LPI.

A system for cryolayer formation and target transport into the laser focus has been developed and tested in a modeL It can operates both as a component of a static positioning system (target on suspender) and for cryotarget positioning by means of injection. The developed target delivery system as well as new methods for cryotarget formation (ILT and FST) constitute the initial technological level of the Cryotarget Factory for ICF reactor. A concept of such a factory is developing which is based on the methods and technical findings obtained at the LPI.
STRUCTURE OF VAPOR DEPOSITED SOLID HYDROGEN CRYSTALS*

Gilbert Collins, Evan Mapoles, Walt Unites, and Tom Bernat
University of California
Lawrence Livermore National Laboratory
P.O. Box 808, Livermore, CA 94551

Solid hydrogen crystals grown slowly through the liquid phase can easily be made several millimeters in extent with flat crystal facets. This tendency to form large crystals with facets makes it difficult to form smooth spherical hydrogen layers for ICF targets. Two ways to prevent large incommensurate facets is to make very small hydrogen crystallites or grow crystals above roughening temperatures.

One way to reduce crystal size is to deposit crystals at low temperature. We report the crystal size and crystal structure of solid hydrogen deposited from the gas phase from the triple point temperature, $T_{tp}$, down to 3 K. $H_2$ and $D_2$ crystals deposited near $T_{tp}$ are millimeter in extent with HCP crystal structure. For deposition temperatures $T_d < 0.4 T_{tp}$, microcrystals are formed with a mixed HCP+FCC crystal phase. This mixed phase transforms into an HCP phase continuously and irreversibly as the temperature increases through 0.5 $T_{tp}$. During this crystal structure transformation the crystallite size increases. Finally we show that flat crystal facets in hydrogen are more stable than previously anticipated. For current NIF target designs, hydrogen crystals near equilibrium will contain facets.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Advanced inertial confinement fusion targets require uniform condensed cryogenic hydrogen fuel layers with inner surface smoothness as low as 1000 Å rms for some designs. For a liquid layer stabilized against gravity, surface tension would provide this surface smoothness. Advanced targets have diameters of 1 to 2 mm, with fuel layers up to 100 μm thick. Current experiments are being conducted on capsules in this size range. Liquid oscillations as well as stable liquid surface configurations have been observed. We will report on our progress towards creating stable uniform liquid layers.

¶Work performed under the auspices of the US Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48 and by W.J. Schafer Associates under Contract DE-AC03-91SF18601.
RECENT RESULTS OF THERMAL GRADIENT LAYERING EXPERIMENTS


TENTH TARGET FABRICATION SPECIALISTS’ MEETING
Taos, NM
Feb., 6-9, 1995

*W.J. Schafer Associates
303 Lindbergh Ave., Livermore, CA 94550

**University of California
Livermore National Laboratory
P.O. Box 808, Livermore, CA 94550
Future ICF experiments will require capsules containing uniform cryogenic layers

- Cryogenic layers can be either liquid or solid.

<table>
<thead>
<tr>
<th>Liquids</th>
<th>Solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>Requires external thermal gradient to achieve centering</td>
<td>(\beta) heating</td>
</tr>
<tr>
<td></td>
<td>- self powered</td>
</tr>
<tr>
<td></td>
<td>- self symmetrizing</td>
</tr>
<tr>
<td>Surface tension should provide a good surface finish</td>
<td>Non-tritiated fluids require external heating source</td>
</tr>
<tr>
<td></td>
<td>Poorer surface finish than liquids</td>
</tr>
<tr>
<td></td>
<td>- May require external source to smooth the surface</td>
</tr>
</tbody>
</table>

There are advantages to liquid layers

- Liquid layers provide more robust, lower convergence ignition designs with somewhat reduced yields
- Control of the liquid layer average temperature allows tuning of the vapor bubble pressure over an 8:1 range
- Specifications for both OMEGA-Upgrade and NIF exceed the measured surface finish in \(\beta\)-layered solid targets
- Liquid layers, if achievable, would provide a smoother surface finish than that obtainable with solid \(\beta\)-layered targets due to surface tension smoothing of the free surface
Thermal gradients induce surface tension driven flows that support liquid layers

- Current experiments have focused on levitating H$_2$ - D$_2$ liquid mixtures
  - Laser heating - direct heating of the capsule wall by light absorption
  - Thermal plate - helium gas column between temperature controlled plates

- A positive (hot on top) thermal gradient is used for a multi-component fluid (i.e. H-D or D-T)
  - The thermal gradient causes preferential evaporation of the lower vapor component from the top of the capsule, generating a concentration gradient
  - The surface tension gradient generated by the concentration gradient opposes gravity and levitates the liquid
The clamped shell experiments were designed to determine if thick layers could be levitated

- In this version a laser was used to heat the top of the capsule
- The capsule was attached to a copper base to maximize the heat flow through the capsule
- One can vary the power and location of the heating by varying the laser power and its position
- This experiment was expected to generate thick but nonuniform layers.
- There was a problem with oscillations and coupling to the control volume
These experiments showed that thick layers can be suspended

- Top layer thicknesses of \(-75 \, \mu\text{m}\) were obtained

- Maximum laser power required to support thick layers was less than 4 mW

- Laser powers of more than 5 mW caused the top layer to evaporate

- The oscillating layer could be stabilized/controlled at laser powers slightly below levels which caused the top layer to evaporate

Calculation of the power absorption vs angle and the associated heat profiles for unmodulated laser heating

Calculated power absorption profile vs angle on the illuminated half of the capsule wall from a flat field source with an effective f-number of \(-15\).

Calculations assume circularly polarized light and a low coherence source to eliminate interference effects.

Temperature profile on a simple capsule model under uniform top heating with non-convective heat flows in the core is shown on right.
The goal of the next set of experiments is to generate uniform layers.
Capsule fill level control is achieved by heating of a liquid filled control volume and a couple of heat activated valves.

Fill Line Heat Switch insures capsule fill cannot change as the vapor pressure changes with increasing average temperature.

Control Volume Heat Switch seals in the liquid isolating the control volume from the room temperature reservoir.

Control Volume operates by using the liquid coefficient of thermal expansion in a sealed cavity to allow accurate control of the capsule fill level.

Average layer thickness is calculated from the estimated volume of the solid near the triple point

The solid volume is approximated by half of the marked ellipsoid of revolution, where \( a \) and \( c \) are the mayor and minor diameters, and half of the sphere zone delimited by the segment \( 2a \) and \( R \) which is the inner radius of the capsule;

thus we obtain:

\[
V_{ld} = \left[ \frac{2}{3} \pi a^2 c + \frac{\pi c^2}{2} (3R-c) \right]^{1.12}
\]

Layer thickness vs. Tave

\( H_2D_2 \) solid bead generated with a 9K/cm gradient in a 1mm plastic capsule. Measured solid ice volume will produce a uniform liquid layer of \(~45\mu m\) average thickness.
Liquid is evident along the side of the capsule

- 50:50 H₂ - D₂
- Thermal gradient = 36 K/cm
- T_{ave} = 21.5 K

The liquid layer shape can be seen to evolve with increasing thermal gradient

- Series of polar plots
- Thermal gradients from 0 - 32 K/cm
- T_{ave} = 21.5 K
- Layer thickness variations diminish with increased gradient
A 2 mm O.D. capsule was used for the next set of experiments

- Oscillations similar to those seen in the clamped shell experiments were readily apparent

50:50 H$_2$ - D$_2$ mixture with a fill corresponding to an estimated 40 $\mu$m uniform layer thickness

- There is some indication that we were able to stabilize a thin (~15 $\mu$m) layer by adjusting the gradient/average temperature $\rightarrow$ oscillations damped out

Summary

- Thick nonuniform layers (~75 $\mu$m) can be supported with the laser driven thermal gradient technique with <4 mW of power

- 25 - 30 $\mu$m thick stable liquid layers can be supported with static thermal gradient of 20 - 30 K/cm
  - Probably adequate, if symmetric, for foam supported liquids with clean 10 $\mu$m inner liquid layer

- Layer shape is dependent not only on the heating profile but the quality of the capsule
  - Capsule wall affects the isotherms seen by the liquid layer

- Oscillations are more prominent in the larger 2 mm capsules
  - Possibly dependent on how the gradient is applied

- Appropriate spatial intensity profiling of the laser will be need to control the shape of the layer
THEORETICAL INSIGHT INTO THE THERMAL-GRADIENT LAYERING OF CRYOGENIC H$_2$-D$_2$

Michael J. Monsler
W.J. Schafer Associates, Inc.
303 Lindbergh Avenue, Livermore, CA 94550

In trying to form a thick uniform layer of a mixture of hydrogen isotopes (such as H$_2$-D$_2$) in an ICF capsule by the application of a vertical thermal-gradient, one hopes to use the surface tension-gradient force to support the cryogenic fluid against gravity. Experimenters have found this to be difficult to accomplish for a thick layer, which requires strong forces. Significant thicknesses of liquid have been levitated, but only in very unsymmetrical or unsteady layers. There are many forces to be balanced, and the fluid has many degrees of freedom in response, only one of which is acceptable. There are gradients in species concentration, temperature, and velocity which must be accurately known to determine the forces. Evaporation and condensation cannot be ignored, because the phase-change dominates the heat-transfer and because the concentration difference between the evaporating and condensing surfaces determines the surface tension-gradient force that lifts the fluid. We must understand these effects in order to control the thermal-gradient-layering process.

Results are presented from three separate analytical models. The first is a steady-state model of a spherically symmetric liquid H$_2$-D$_2$ layer in the preferred flow pattern. We give the predicted operating characteristics for Omega-Upgrade and NIF capsules. The operating characteristic is a plot of the temperature difference across the shell versus the heat-transfer through the shell, required to achieve a uniform layer. The second model gives an explanation of a pulsating mode observed in thermal layering experiments. Results are given for the expected rate of pulsation, and the key controlling parameters are discussed. The third model is an explanation of a correlation of experimental data first suggested by J. Sanchez of LLNL. We explain why the temperature gradient divided by the layer thickness should have the observed magnitude and dependence on the average capsule temperature, quite independently of other capsule parameters. In this presentation, we emphasize how lessons learned from analytical modeling of the dominant physical processes can help to control thermal-gradient layering in the laboratory.

Work supported by U.S. Department of Energy under Contract No.DE-AC03-91SF18601.
Thermal Gradient Layering of Cryogenic H₂-D₂

Dr. Michael Monsler

W. J. Schafer Associates, Inc.
303 Lindbergh Avenue
Livermore, CA 94550

10th Target Fabrication Specialists' Meeting
Taos, NM

Feb. 6-10, 1995
We have developed a nonlinear model to calculate the flowfield and forces in a thick liquid-layer ICF target.

The model determines the thermal conditions \( (T, \frac{dT}{dz}, Qdot) \) for which the net force on a symmetric layer vanishes, in a volume integrated sense.

- The model works for pure \( D_2 \) or \( H_2-D_2 \)
- The model cannot handle unsteady or asymmetric conditions
- The model is used to predict the conditions needed to support liquid layers in Omega-Upgrade and NIF capsules, with and without the presence of foam.

1. **Conservation of Mass** - Exact velocity profiles are derived which satisfy all boundary conditions, but have unknown parameters.

2. **Conservation of Momentum** - The integral form of the momentum equation is used, with the parameterized velocity profiles, to generate an algebraic equation for the net force on the layer as a function of the parameters. The equation is solved iteratively for the \( \Delta T \), across the upper bubble that causes the force to vanish.

3. **Conservation of Energy** - We account for all the heat flows through the vapor, the liquid, and the shell, corresponding to the \( \Delta T \) across the bubble.

4. **Simplifying Assumption** - Assume that the unknown surface tension gradient term \( \frac{d\sigma}{dT} \) can be derived from experimental data.
This nonlinear model is capable of handling the thick layer expected in Omega Upgrade and NIF targets.

Case 2: Omega Upgrade

- Linearized theories are only valid up to Reynolds numbers of unity
- Reynolds numbers exceed unity for layers thicker than 30 µm.

The surface-tension-gradient is a complicated and unknown function of the boundary conditions.

- The surface-tension-gradient in H\textsubscript{2}-D\textsubscript{2} (or DT) is positive

\[
\frac{d\sigma}{dT} = \left[ \frac{\partial \sigma}{\partial T} + \sum_i \frac{\partial \sigma}{\partial x_i} \right] \frac{dT}{dz}
\]

- But \(\frac{d\sigma}{dT}\) (as a property of the fluid) is always negative, even for multicomponent mixtures of hydrogen, so it cannot be a local property of the fluid.

- \(\frac{d\sigma}{dT}\) must be calculated by balancing diffusion and evaporation/condensation, for both components at both interfaces.

- Until this is done correctly, we must scale from what little experimental data we have for thin layers.
The model is fit to the thin-layer results of Kim et al. obtained in a helium cell.

We provide results of calculations for five cases. All are for H$_2$ - D$_2$ @ 20 K.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Case 1 &quot;Kim H$_2$-D$_2$&quot;</th>
<th>Case 2 &quot;Omega Upgrade&quot;</th>
<th>Case 3 &quot;Omega-U Foam&quot;</th>
<th>Case 4 &quot;NIF&quot;</th>
<th>Case 5 &quot;NIF Foam&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capsule</td>
<td>Glass</td>
<td>Polymer</td>
<td>Polymer</td>
<td>Polymer</td>
<td>Polymer</td>
</tr>
<tr>
<td>Outer radius, $\mu$m</td>
<td>300</td>
<td>461</td>
<td>461</td>
<td>1110</td>
<td>1110</td>
</tr>
<tr>
<td>Shell thickness, $\mu$m</td>
<td>6.25</td>
<td>6</td>
<td>6</td>
<td>160</td>
<td>160</td>
</tr>
<tr>
<td>Inner radius, $\mu$m</td>
<td>293.75</td>
<td>455</td>
<td>455</td>
<td>950</td>
<td>950</td>
</tr>
<tr>
<td>H$_2$-D$_2$ Filled Foam</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Outer radius, $\mu$m</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>950</td>
</tr>
<tr>
<td>Foam thickness, $\mu$m</td>
<td>0</td>
<td>0</td>
<td>58</td>
<td>0</td>
<td>60</td>
</tr>
<tr>
<td>Inner radius, $\mu$m</td>
<td></td>
<td></td>
<td></td>
<td>397</td>
<td>890</td>
</tr>
<tr>
<td>Free H$_2$-D$_2$ Liquid</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Outer radius, $\mu$m</td>
<td>293.75</td>
<td>455</td>
<td>397</td>
<td>950</td>
<td>890</td>
</tr>
<tr>
<td>Layer thickness, $\mu$m</td>
<td>6.85</td>
<td>78</td>
<td>20</td>
<td>80</td>
<td>20</td>
</tr>
<tr>
<td>Inner radius, $\mu$m</td>
<td>286.90</td>
<td>377</td>
<td>377</td>
<td>870</td>
<td>870</td>
</tr>
</tbody>
</table>
The composition of the vapor and liquid phases depends on the fill conditions as well as temperature.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Case 1</th>
<th>Case 2 &amp; 3</th>
<th>Case 4 &amp; 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capsule Volume, m³</td>
<td>1.06 x 10⁻¹⁰</td>
<td>3.95 x 10⁻¹⁰</td>
<td>3.59 x 10⁻⁹</td>
</tr>
<tr>
<td>Fill pressure @ 300 K, atm</td>
<td>75</td>
<td>600</td>
<td>280</td>
</tr>
<tr>
<td>Fill mass, kg</td>
<td>9.35 x 10⁻¹⁰</td>
<td>2.1 x 10⁻⁸</td>
<td>1.05 x 10⁻⁷</td>
</tr>
<tr>
<td>Composition of fill @ 300 K</td>
<td>50% H₂ - 50% D₂</td>
<td>50% H₂ - 50% D₂</td>
<td>50% H₂ - 50% D₂</td>
</tr>
<tr>
<td>Composition of vapor @ 20 K</td>
<td>73.2% H₂ - 26.8% D₂</td>
<td>75.2% H₂ - 24.8% D₂</td>
<td>74.9% H₂ - 25.1% D₂</td>
</tr>
<tr>
<td>Composition of liquid @ 20 K</td>
<td>47.1% H₂ - 52.9% D₂</td>
<td>49.7% H₂ - 50.3% D₂</td>
<td>49.3% H₂ - 50.7% D₂</td>
</tr>
<tr>
<td>Fractions of liquid and vapor</td>
<td>88.9% liq - 11.1% vap</td>
<td>99.9% liq - 1.1% vap</td>
<td>97.2% liq - 2.8% vap</td>
</tr>
</tbody>
</table>

Thick and thin layers behave differently with respect to composition and layer thickness.

Case 1: Kim H₂-D₂

Case 2: Omega-Upgrade
The liquid layer is supported about equally by the surface-tension-gradient force at the interface and viscous shear at the wall.

Case 2: Omega Upgrade

The operating characteristics for Cases 2 and 3 Omega-Upgrade capsules with and without foam
The operating characteristics for Cases 4 and 5 NIF capsules with and without foam

The temperature differences needed across the bubble are extremely small compared to those across the capsule.

<table>
<thead>
<tr>
<th>Case 2: Omega-Upgrade</th>
<th>Case 4: NIF</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \Delta T_{\text{shell}} = 0.022 \text{ K} )</td>
<td>( \Delta T_{\text{shell}} = 1.371 \text{ K} )</td>
</tr>
<tr>
<td>( \Delta T_{\text{liq}} = 0.038 \text{ K} )</td>
<td>( \Delta T_{\text{liq}} = 0.084 \text{ K} )</td>
</tr>
<tr>
<td>( \Delta T_{\text{vap}} = 6.4 \times 10^{-5} \text{ K} )</td>
<td>( \Delta T_{\text{vap}} = 1.67 \times 10^{-4} \text{ K} )</td>
</tr>
<tr>
<td>( \Delta T_{\text{tot}} = 0.12 \text{ K} )</td>
<td>( \Delta T_{\text{tot}} = 2.91 \text{ K} )</td>
</tr>
</tbody>
</table>
The heat flow distribution must be carefully controlled to obtain the desired flow pattern.

Conclusions

1. One current models indicate that liquid H₂-D₂ layers of 50 - 100 μm thickness should be supportable with the following parameters:
   - Omega-Upgrade: ΔT < 0.2 K and Q < 0.3 mW
   - NIF: ΔT < 4 K and Q < 5 mW

2. The higher requirements for NIF over Omega-Upgrade are fully explained by the ratio of liquid masses to be lifted (x 3.55) and the ratio of shell thicknesses (x 26.6).

3. Immobilizing 75% of the liquid in a foam, leaving a 20 μm layer of free liquid, reduces the required heat transfer rate by 60 - 70%, but the required temperature difference is not reduced as much, largely because heat conduction in a stagnant liquid in foam is poor compared to that in a convecting liquid.

4. There is enough lifting force at reasonable heat flow rates for thermal-gradient-layering to be successful, particularly for Omega-Upgrade. Control over the heat flow/temperature distributions is the critical issue.
General Atomics is developing a transfer cryostat that will permit gas cooled inertial confinement fusion (ICF) targets to be transferred cryogenically (T < 20 K) from a cryostat where they are filled to a cryostat where they will be layered and inserted into the target tank of OMEGA-Upgrade ICF facility at the University of Rochester's Laboratory for Laser Energetics. The transfer cryostat's bottom sections will be resealed inside of the fill cryostat and vacuum reestablished between the sections. Vacuum seals have been developed that allow resealing while at cryogenic temperatures. A number of designs and materials were investigated. The best designs utilized copper gaskets plated with one to two thousands of an inch of indium, a hemispherical ridge flange, and a solder flange. The median leak rate of this seal was found to be $2 \times 10^{-9}$ mbar l/sec for differential helium pressure of 100 Torr and temperature 15 K. The median was computed for a seal continuously kept at 15 K while being unsealed and resealed. During one test run, a seal was successfully resealed 11 times before the test apparatus warmed up.

The seal developed easily allows for the vacuum spaces of the transfer cryostat to be evacuated with a modest pump, once the bottom sections have been resealed.
CRYOGENICALLY RESEALABLE VACUUM SEALS

NEIL B. ALEXANDER

This work supported by the United States Department of Energy under contract No. DE-AC03-91SF18601.
Why do we need resealable vacuum cryoseals?

Seal requirements

Test apparatus

Seal types tested and test results

Application of seals in ICF target cold transfer

GENERAL ATOMICS

RESEALABLE CRYOSEALS NEEDED FOR PERMEATION FILLED, HIGH DENSITY D-T PLASTIC ICF TARGETS

Separate, isolated fill station used for permeation filled, high density ICF targets
- High pressures and large D-T inventories dictate isolated fill station
- Don't want accident contaminating large, expensive facilities

Cold Transfer Cryostat moves target from fill station to Fusion Facility or further processing sites
- Target's fuel always kept condensed or else target ruptures

Resealable cryoseals allow targets to be moved into and out of Transfer Cryostat inner chamber
- D-T self heating and target mount's low thermal conductivity dictate helium gas cooling
CRYOSEAL REQUIREMENTS

- Must reseal at temperature of 15 K ± 5 K
- Must prevent helium from thermally shorting cryostat
  - differential pressure 1 to 10 torr
  - minimum leak rate
    - Actively pumped cryostat: \(-10^{-5}\) mbar l/sec (design dependent)
    - Intermittently pumped cryostat: \(-10^{-8}\) mbar l/sec (design and time req.'s dependent)
- Least effort to reseal and replace
  - Sealing is remote operation; Inside another cryostat
- Number of seal makes and breaks required at low temperature
  - Minimum Case: 1 make, then 1 break
  - Convenient Case: 2
  - Ideal Case: Multiple
- Robust

SEAL TYPES TESTED

- Gasket seals between flat face and blunt nose flanges
  - Teflon, OFHC copper, indium plated OFHC copper, platinum, stainless steel, silver plated stainless steel, nickel, silver plated nickel

  ![Gasket seals diagram]

- Spring loaded C-ring in both radial and face configurations
  - Silver, aluminum, Teflon, Kel-F, polymer filled Teflon, glass filled Teflon, polyethylene
  - Manufacturers: Helicoflex, Bal-Seal, American Variseal

  ![C-ring diagram]
TEST FIXTURE FOR A CRYOGENICALLY RESEALABLE VACUUM SEAL

- 9 Seal designs tested
- 36 design / material combinations tested
- Optimization of indium plating
- Sealing procedure investigated

Made From 1/2" Cajon VCR, 316 Stainless steel

Thread: 7/8 – 16
Seal Ø 1/2

NOT TO SCALE
SUMMARY OF CRYOGENIC VACUUM RESEAL TESTS

- Flat Gasket, Round Ridge Faces
- Flat Gasket, Flat Faces
- Soldered Gasket, Other Face Round Ridge
- Keyed Soldered Gasket, Other Face 90° Ridge
- Face Seal with C-ring, Polymer
- Face Seal with C-ring, Metal
- Radial Seal with C-ring, Polymer
- Radial & Face Seal with C-ring, Polymer
- Radial Seal with C-ring, Metal
- Soldered Gasket, Other Face 90° Ridge
- Initially Sealed at 15 K

TEMPERATURE: 15 – 295 K
DIFFERENTIAL HELIUM PRESSURE: 100 TORR
SUMMARY OF CRYOGENIC VACUUM INITIAL SEAL TESTS

TEST RESULTS FOR BEST PERFORMING RESEALABLE CRYO-SEALS

- Seal opened and closed at 15 K
- Differential helium pressure 100 Torr
- Seal A: OFHC Copper gasket plated with 0.002" indium, soldered with indium to one flange, other flange hemispherical ridge
- Seal B: OFHC Copper gasket plated with ~1/4 thousands inch indium, both flanges hemispherical ridges
INDIUM COATING THICKNESS OF COPPER GASKET OPTIMIZED

![Graph showing Indium Thickness vs Median Leak Rate](image)

- Flat Gasket, Round Ridge Faces
- Soldered Gasket, Other Face Round Ridge
- Soldered Gasket, Other Face 90° Ridge

TEMPERATURE: 15 K
DIFFERENTIAL HELIUM PRESSURE: 100 TORR

OVERVIEW OF COLD TRANSFER CRYOSTAT

![Diagram of Cold Transfer Cryostat](image)
COLD TRANSFER CRYOSTAT ASSEMBLY

PRECISION BELLOWS MANIPULATORS

- FOR COLD TRANSPORT CRYOSTAT
  Z: 37", X & Y: ±0.5"

- FOR CRYO-WRENCH
  Z: 36"

GENERAL ATOMICS
PERMISSIBLE LEAK RATE IN COLD TRANSFER CRYOSTAT

- Volume of Inner Shroud (T = 20 K) 43 cc
- Total Volume of Outer Shroud (T = 50 K) 1,038 cc
- Volume of Bellows 5,000 cc

- Typical Leak Rates of $\frac{1}{4}$" Cryoseals = $2 \times 10^{-9}$ mbar l/sec
  - Assume leak 1000x larger, seal dia. $1\frac{1}{2}$".
  - Leak Rate from inner shroud at 1 torr is $5.3 \times 10^{-8}$ mbar/sec

- Assuming $10^{-3}$ mbar in outer shroud, after one day, the pressure rises to $5.6 \times 10^{-3}$ mbar

- For a Mean Free Path Greater than $\lambda = 3$ cm $\Rightarrow P \leq 5.6$ mbar, gas conduction is in a low pressure regime, thermal conductance is given by

$$\frac{Q}{A} = 0.7 \sqrt{\frac{T_{\text{hot}}}{T_{\text{cold}}}} P (T_{\text{hot}} - T_{\text{cold}})$$

- The Heat Load After One Day Rises from 0.5 Watt to 2.1 Watt
- This is Much Less than the Available Cooling Power of 9 Watts at 20 K
CONCLUSIONS AND FUTURE

- Prototype cryogenically resealable vacuum seals have been successfully developed to a useful level.
- Scaled up seals will be implemented and tested in a full scale target fill and transfer apparatus within a year.
A sapphire microwave cavity has been designed for symmetrization of a solid fuel layer of an ICF target by plasma heating. The cavity will resonate at 3 GHz in TM_{010} mode. With the cavity coated with copper, the power dissipation is calculated to be less than 0.02 Watts at 15°K and the electric field at the center of the cavity is 1000V/cm. The Q of the cavity at 15°K is calculated to be 2\times10^5.

The correctness of the above design has been verified by performing measurements on a cavity having similar dimensions filled with HiK powder. The resonant frequencies are calculated to be 2.93 and 4.67 GHz for the lowest and the second modes, respectively. A network analyzer measurement shows that the lowest mode has a frequency of 2.95 GHz, in good agreement with the calculated value. Excellent impedance matching is shown at an antenna length of 0.485cm.

The plasma heating uniformity of a 3mm quartz shell has been observed with an infrared camera. The shell was positioned in a copper cavity resonating at 2.96 GHz. The density of D_2 inside the shell was 2.8g/m^3, equivalent to the equilibrium vapor pressure of D_2 at 12°K. The heating uniformity increases with a smaller shell due to the increasing dominance of the diffusion effect. Because of this, one can increase gas density, if desired, while maintaining the heating uniformity. This may allow the measurement to be performed with a high gas pressure inside the shell.
Design of a Resonant Cavity for Plasma Heating of Solid D$_2$ or DT Fuel Layers

C. M. Chen, H. Kim, M. Wittman, and S. Letzring
University of Rochester
Laboratory for Laser Energetics

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Taos, New Mexico
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Summary

A resonant cavity for plasma heating solid D\textsubscript{2} or DT fuel layers has been designed.

- Symmetrization of a solid D\textsubscript{2} or DT layer may be achieved by plasma-induced uniform heating of its inner surface. This technique is especially useful for D\textsubscript{2}, which cannot be β-layered.

- The plasma is produced by placing the capsule inside a resonant cavity that is driven by an RF oscillator. The capsule must be located in a region where the electric field is uniform.

- Preliminary experiments were performed using a 3-mm-diam quartz shell in a room-temperature resonant cavity to determine the conditions necessary to produce a uniform temperature distribution on the shell's surface.

- Results of these experiments were used to design a sapphire resonant cavity for use in experiments to be conducted at cryogenic temperatures.

Symmetrization of a solid D\textsubscript{2} or DT layer may be achieved by plasma-induced uniform heating of the inner surface.
Uniform heating may be achieved by applying a high-frequency electric field with a low field strength.

\[ \frac{Q_\parallel}{Q_\perp} = 1 + \frac{\mu_e eA E}{\mu_i \pi k T_e} \]

\[ A = \frac{E}{\sqrt{\frac{1}{\mu_e} \omega^2 + \left( \frac{\omega}{e/m} \right)^2}} \]

The cavity is designed to produce a uniform electric field in the central region.
The resonant cavity is designed using a computer program called "Superfish".

Superfish calculates the resonant frequencies and the properties of the resonant modes by solving the 2-D Helmholtz equation.

- TM modes

\[ \frac{1}{r} \frac{\partial}{\partial r} \left( r \frac{\partial H_\theta}{\partial r} \right) + \frac{\partial^2 H_\theta}{\partial z^2} + \varepsilon \omega^2 \frac{\partial^2 H_\theta}{\partial t^2} = 0 \]

- Transverse electric fields

\[ E_r = \frac{1}{\varepsilon \omega} \frac{1}{r} \frac{\partial}{\partial z} (r H_\theta) \]

\[ E_z = \frac{1}{\varepsilon \omega} \frac{1}{r} \frac{\partial}{\partial r} (r H_\theta) \]

The temperature distribution inside the 3-mm quartz shell is measured with an infrared imaging system.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency</td>
<td>3.0 GHz</td>
</tr>
<tr>
<td>Pulse width</td>
<td>25 μs</td>
</tr>
<tr>
<td>Duty cycle</td>
<td>2.5%</td>
</tr>
<tr>
<td>D₂ density</td>
<td>16.65 μg/cm³</td>
</tr>
</tbody>
</table>
Although the plasma radiation is not uniform, the temperature is uniform for D$_2$ densities less than 16.5 $\mu$g/cm$^3$.

The heat generated has been calculated at several D$_2$ densities.
A sapphire resonant cavity was designed for cryogenic experiments.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resonant frequency</td>
<td>2.996 GHz</td>
</tr>
<tr>
<td>Electric field at the cavity's center</td>
<td>1000 V/cm</td>
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<tr>
<td>Stored energy</td>
<td>278 nJ</td>
</tr>
<tr>
<td>Power dissipation</td>
<td>2.84 W at 293°K</td>
</tr>
<tr>
<td></td>
<td>22 mW at 15°K</td>
</tr>
<tr>
<td>Q factor</td>
<td>1840 at 293°K</td>
</tr>
<tr>
<td></td>
<td>238,000 at 15°K</td>
</tr>
</tbody>
</table>

The proposed sapphire cavity is a two-part assembly with a spherical void.
A cavity filled with HiK powder was constructed and the resonant frequencies were measured.

Antenna (4.85 mm long)

High dielectric constant material (HiK powder, ε = 9.0)

Copper body

Measured frequencies of the powder-filled cavity agree with calculated values.

1st resonant frequency: 2.94 GHz

2nd resonant frequency: 4.67 GHz

1: 2.95 GHz
2: 4.63 GHz
3: 4.84 GHz
Conclusions

A resonant cavity for plasma heating solid D\textsubscript{2} or DT fuel layers has been designed

- The "Superfish" program was successfully used to model the electrical characteristics of both the room-temperature and cryogenic resonant cavities.

- Preliminary experiments performed using a 3-mm-diam quartz shell in a room-temperature resonant cavity indicate that, although the visible radiation from the plasma is not uniform, the temperature of the quartz shell's surface is uniform for D\textsubscript{2} densities less than 16.5 \(\mu\text{g/cm}^3\).

- Results of these experiments were used to design a sapphire resonant cavity for use in experiments to be conducted at cryogenic temperatures.

- Since the sapphire cavity could not be machined, a copper cavity filled with high-dielectric-constant material was used to verify the sapphire cavity design.
We shall present the capability of our target laboratory responsible for providing all the target required by the experimental laser program at CEL-V.

A variety of plane, spherical and complex targets are produced for experiments with Phebus, Octal and P 102, in addition our program includes basic research and development usable for advanced target fabrication.

A summary of new typical areas now routinely used is as follows:

- **Plastic shell production and gas filling**
The polystyrene (PS) shell production is based on the microencapsulation process. Pure argon or a mixture deuterium-argon is used to fill the ball. Thickness and pressure measurement are done, using interferometry, X-ray absorptiometry with a scanning electron microscope and dew point technic.

- **Coating layers and low density materials studies.**
The Laboratory uses several coating techniques for a variety of metallic and polymeric elements, some in pure form others as compounds. Low density foams are prepared from an inverse emulsion process. Density as low as $3 \times 10^{-2}$ g.cm$^{-3}$ have been obtained and by high speed machining various shapes can be produced.

- **Influence of micromachining on advanced target fabrication.**
By coupling micromachining and vacuum deposition x-ray point source $20 \mu m$ in diameter are obtained, the source can be made of different materials as for example rare earths (praseodymium, erbium, terbium,...).

With reactive ion etching or excimer laser it is possible to produce throught a mask, sinusoidal profile on flat polymer substrate. The period is around $60 - 100 \mu m$, with $0,2 - 5 \mu m$ of amplitude.

Stylus profilometer, white light interferometry or atomic force microscope are used for characterization.

- **Several complex targets.**
About one thousand targets have been prepared for the experimental program in 1994. Among many structure we will present the indirect drive structure, a type of cavity for spectroscopic measurement and a target for absorption spectroscopy studies.

- **Preliminary studies on cryogenics targets.**
In the area of cryogenic target, we have investigated a series of experiment relation with a foam target design, wetted with liquid deuterium. We used the capillarity process to fill the foam and the main results on this experiment are given.
ICF TARGET TECHNOLOGY IN RUSSIAN FEDERAL NUCLEAR CENTER

We give the results of the production process for fabrication of targets to be used in experiments on the laser "ISKRA-4" and "ISKRA-5" system. The main types of targets used in experiment share direct drive targets, inverted corona targets, and indirect drive targets.

The production techniques have been developed and hollow glass and polystyrene microspheres are fabricated and filled with gaseous D-T mixture, neon or argon. For a diagnostic purpose the microspheres' glass is doped with europium. Microspheres are coated with polymeric and metallic films.

We have developed the techniques of fabrication of thin-wall metallic semi-spheres and solid films containing deuterium and tritium.

Microscopy, microinterferometry, and x-ray examination are used as routine methods for the target parameters measurement. In addition, optical methods with increased sensitivity to transparent film inhomogeneities have been developed; these are the multibeam interferometry and holographic interferometry. For recording the integrity and homogeneity of the films containing tritium we used the autoradiography. The film composition is investigated using an optical infrared spectro-photometer, a massspectrometer, a x-ray microanalyses an Auger spectrometer.

A series of investigations has been performed on the cryogenic target technology, automatic selection and control of microspheres with geometrical parameters, and microsphere precise rotation.
ICF TARGET TECHNOLOGY AT RUSSIAN FEDERAL NUCLEAR CENTER.


Russian Federal Nuclear Center, VNIIEF, Arzamas-16, Russia.

The main efforts of the body working in the area of the ICF target fabrication are directed at ensuring the experiments performed on the “ISKRA-4” and “ISKRA-5” laser system. The main types of targets used in experiments are direct drive targets, inverted corona targets, and indirect drive targets.

The direct drive target is a glass spherical container coated with a metal or polymeric film and filled with a D-T mixture and some diagnostic gas\(^1,2\). The inverted corona target is a spherical shell with holes for introducing laser radiation. The inside surface of the shell is coated with a substance containing heavy hydrogen isotopes\(^3,4\). The indirect drive target is assembled from a spherical shell with holes for introducing laser radiation and the direct drive target placed in the shell center. The inside surface of the shell is coated with a large atomic number substance\(^5\) (Fig.1).

For production of the direct drive target, the manufacturing techniques have been developed and hollow glass and polystyrene microspheres are fabricated. Hollow glass microspheres are fabricated by free-fall of liquid glass drops or dry gel in a four meter vertical kiln similar to Ref. 12. Polystyrene microspheres are fabricated from polystyrene particles with a blowing agent in the vertical kiln too. These methods allow to manufacture glass microspheres with diameters from 50 \(\mu\)m to 1 mm and wall thicknesses from 0.5 to 10 \(\mu\)m, and aspect ratios from 20 to 500. The microspheres are suitable for use in experiments, that is they have a thickness inhomogeneity less than 5% and nonsphericity less than 1%. Polystyrene microspheres are fabricated with diameters up to 800 \(\mu\)m and wall thicknesses from 1 to 10 \(\mu\)m.

Target filling with a gaseous D-T mixture is performed by diffusion in the presence of a high temperature and external pressure up to 200 atmospheres. Addition of the diagnostic neon gas to the D-T mixture is performed by diffusion too. The target manufacturing technique has been developed with addition of gaseous argon to the D-T mixture. We make a hole of the size less than 10 \(\mu\)m in a hollow microsphere by a laser beam /see for example Ref. 13/. Then the hole is sealed within a special chamber filled with gaseous argon. The chamber construction allows to evacuate it, fill with argon to the pressure of one atmosphere, to orientate and seal the target and the hanging filament, and view the target in three perpendicular to each other directions (Fig. 2).

We and Miyanaga with coworkers\(^14\) have independently proposed doping the shell with europium for the collector calibration used in the radiochemical method of measuring \(\rho \cdot \Delta R\) and \(\rho \cdot R\) in a moment of the largest target compression. Europium has a large cross section of the thermal neutrons activation and is used as a marker substance. The developed method of the glass doping ensures the inhomogeneity of the europium amount in different microspheres and its distribution in the glass volume of a separate microsphere less than 15%. With the \(\text{Eu}_2\text{O}_3\) weight concentration of 2%, the microsphere exposure to the neutron flux of \(2 \times 10^{17}\) \(\text{cm}^{-2}\) gives the intensity of \(\gamma\)-peak of the activated isotope \(^{152}\text{Eu}\) radiation exceeding the measurement sensitivity by a factor of 200. In addition, the time interval between irradiation and measurement was 20 hours. In Fig. 3 the Eu concentration is given at different points along the microsphere equator. The distribution of its X-ray response on the microsphere surface with an area of 40x50 \(\mu\)m\(^2\) is shown too.

Gas filled glass targets are coated with polymers and metals. There have been developed techniques for depositing poly(paraxylilene) coatings of a thickness up to 50 \(\mu\)m and metal films with a thickness from 1 to 10 \(\mu\)m depending on a class of metal. The nonuniformity of layer thickness is smaller than 5%.
For fabrication of the inverted corona targets, the techniques of manufacturing solid layers containing heavy hydrogen isotopes (beryllium and titanium hydrides and polyethylene) have been developed. Beryllium deuteride or deuterotritide is deposited by evaporation of metallic beryllium in a low-pressure deuterium or deuterium-tritium atmosphere and by deposition on the cooled substrate surface.

A film of deuterated polyethylene was formed by laser evaporation of a polyethylene pellet in vacuum. A technique has been developed for deposition of polyethylene coating from a solution.

An investigation of the material layers stability against decomposition by β-radiation of the tritium shows that in contrast to beryllium hydride DT-polyethylene decomposes very rapidly. In Fig. 4 the temporal dependence is represented for a counting rate of secondary X-ray pulses caused by passing of β-electrons from the BeDT layer in the copper substrate. In the second graph the temporal dependence of the activity of products formed by thermal decomposition of DT-polyethylene samples is shown. The solid lines represent the natural loss of tritium because of radioactive decay.

In an investigation designed to reveal impurities in a film we found a considerable concentration of oxygen in the surface layer of the titanium hydride. In Fig. 5 a ratio of amplitudes of the Auger peaks for oxygen and titanium is shown against the duration of argon-ion etching of titanium deuteride and metallic titanium films. The solid curves are dependences of the type $Y = A + B \cdot \exp(-t/T)$ plotted by the least-squares method.

Microscopy, microinterferometry and X-ray examination are used as routine methods for measurement of geometrical target parameters (diameter, wall thickness, thickness nonuniformity). We use optical interferometry for the measurement of the D-T mixture amount inside transparent shells. The accuracy of measurement by optical methods is ±1 μm for the diameter, ±0.05 μm for the wall thickness, 5% for the thickness nonuniformity, and ~3 atm. for the gaseous D-T pressure with the microsphere diameter of 150 μm. The accuracy of pressure measurement increases with the diameter increase. The argon amount is measured by measuring the intensity of characteristic X-ray radiation generated by β-electrons of tritium, similar to Ref. 15. The accuracy of measurement of the geometrical parameters of multilayer spherical targets by X-ray examination is ±0.5 μm. Smoothness of the target surface is determined by viewing it through an optical or scanning electronic microscope (see Fig. 6, 7).

The average thickness of the films containing fuel in inverted corona targets is determined by measuring the film thickness on a flat specimen formed simultaneously with the spherical layer. For layers containing tritium the distribution of thickness and the integrity of the film on half-shells is studied by autoradiography. The accuracy of measurement is 50%. The images of half-shells of the inverted corona target with a BeDT layer are given in Fig. 7. They are obtained by exposure to β-electrons of tritium with the aid of a pinhole camera. One of autoradiograms revealed the damage of the film.

The amount of tritium contained in a layer of half-shells and microspheres is deduced from the intensity of secondary X-rays generated by the passage of β-electrons across the film and substrate. The ratio of the amounts of the hydrogen isotopes is found by thermal decomposition of the material and subsequent analysis of the gaseous phase with a mass spectrometer. Moreover, for transparent specimens recording and analysis of infrared optical absorption spectra are used. The errors of all these methods are in the range of 10-50%. An investigation of the surface layer composition is performed by an X-ray microanalyser and Auger spectrometer.

Optical methods with increased sensitivity to transparent specimen nonuniformities have been developed and used. These are the multibeam interferometry and holographic interferometry. The multibeam interferometry special feature is a small width of the interference fringe. This allows to
measure the fringe displacement with an accuracy up to 1/300. Besides, the error of the wall thickness measurement decreases to 0.002 µm, and the error of the measurement of the gas pressure inside a microsphere decreases to ~0.1 atmosphere at the diameter of 150 µm (Fig. 8). Holographic interferometry is used mainly in cryogenic targets investigations. It allows to observe a change in the cryogenic layer thickness and to decrease the measurement error by a factor of ~4 in comparison with the routine interferometry. Holographic interferometer is used mainly in cryogenic targets investigations. It allows to observe a change in the cryogenic layer thickness and to decrease the measurement error by a factor of ~4 in comparison with the routine interferometry.

References.

Fig. 1 TARGETS FOR LASER "ISKRA-4" AND "ISKRA-5" SYSTEM

Direct drive target

- D-T+Ne (Ar)
- Glass microsphere
- Polimerous (metallic) coating

Inverted corona target

- Solid coating contained D-T
  - BeD$_2$
  - BeDT
  - TiD$_x$
  - TiD$_7$Ti$_{17}$ (CD$_2$)$_n$ (CD,Ti$_{10}$)$_n$

Indirect drive target

- Cu
- Large Z material (Au)
Fig. 2 CHAMBER FOR MICROSPHERES FILLING WITH ARGON
The target glass is doped with europium (concentration 1-2 %)
Collectors gather the product of the target disintegration
Collectors are calibrated by europium activated with thermal neutrons
\[ ^{151}\text{Eu} (n, \gamma) ^{152}\text{mEu} \quad (\sigma = 1400 \pm 300 \text{ b}) \]
The intensity of the \(^{152}\text{mEu} \gamma\) -peak exceeds the detector sensibility by a factor of 200 in 20 hours after the activating \((\Phi_{\text{therm.neutr.}} = 2 \times 10^{17} \text{ cm}^{-2})\).

Distribution of europium over microsphere surface
over an equator over an area 40 * 50 \(\mu\text{m}^2\)
Fig. 4  DECOMPOSITION OF THE FILM MATERIAL CONTAINING TRITIUM

Rate of tritium loss out of film

Relative counting rate of pulses from a gas-discharge counter, obtained for a half-shell coated by BeDT, plotted as a function of the time from moment of the film preparation.

Relative activity of the gas formed by thermal decomposition of DT-polyethylene samples, plotted as a function of the time from moment of their preparation.

The solid lines represent the natural loss of tritium because of radioactive decay.
Ratio of the amplitudes of the Auger peaks of oxygen and titanium, plotted as a function of the duration of argon-ion etching of titanium deuteride and metal titanium. The solid curves are dependences of the type $Y = A + B \exp(-t/T)$ plotted by the least-squares method.
Fig. 6

METHODS FOR TARGET CHARACTERIZATION

Geometrical parameters

Microscopy

X-ray examination

Optical microinterferometry

Amount of the gas in microsphere

Optical interferometry (D-T)

Examination of characteristic X-ray (Ar)

Layer thickness

Test flat specimen

Autoradiography

Interferometric profile measurement

Composition and smoothness of shell surface

Secondary X-ray generated by β-electrons of tritium

Amount of tritium and deuterium

Optical infrared spectrophotometer

Mass spectrometer

X-ray microanalyses

Auger spectrometry

Optical and electronic microscopy

Measuring activity of thermal decomposition gaseous productions
Methods for Target Characterization

Routine methods for target geometrical parameters measurement:

- X-ray examination
- Autoradiography

Image of half-shell obtained by exposure to β-electrons of BeDT film with the aid of a pinhole camera.

Parameters:

- ΔR = 1 μm, Δd = 0.05 μm, Δd/d = 5%, Δp = 3 atm
- ΔR = 0.5 μm
MULTIPLE-BEAM INTERFEROMETRY

Experimental scheme
Method of two exposure
Interferogram of cryogenic layer

Holograms reconstruction in holographical interferometer
Interferograms of cryogenic layer with sensitivity increase $M$
A range of experimental targets have been produced for a feasibility study of Hugoniot EOS measurements on Helen.

Aluminum was chosen as the standard material and the targets have ranged from witness plate to double steps with pre-heat shields.

More complex targets using aluminum and copper steps have been used together with some targets coated with parylene C.

The problems associated with the manufacture and characterization to the accuracy required for these experiments will be detailed. These include manufacture and fabrication of targets with flat substrates, particularly for the multiple step experiments. Proposals for improving future targets will also be given.

The current results will be given where 2-4% accuracy has been achieved for the shock velocities in copper and parylene steps.
Occasionally target designs require the presence of diagnostic dopants to facilitate temperature measurements. To this end polymethylpentene (PMP or TPX) foams were produced with very low densities (3 to 5 mg/cc) and low levels of diagnostic dopants. The dopants added to the foams were chlorine (Cl), titanium (Ti), chromium (Cr), and manganese (Mn). Chlorine foams were made with levels as high as 5 wt% chlorine, while the metal doped foams were produced with metal loadings as high as 1 wt%.

The average foams densities were determined using $\beta$-transmission, and the dopant amounts were determined using x-ray fluorescence. Procedures for doped foam production and measurements of the resulting foam characteristics will be presented.
Production of Metal Doped Low Density Foams

Mike Mitchell
Pete Gobby
Norm Elliott

MST-7
**Goal:**

Produce low density (<5 mg/cc) carbon/hydrogen foam doped with diagnostic metals.

Metals: Titanium (Ti), Chromium (Cr), and Manganese (Mn)

Foam: poly(4-methyl-1-pentene) a.k.a. PMP or TPX

\[
\begin{array}{c}
\text{CH}_2 \\
\text{CH}_2 \\
\text{CH}_3 \\
\text{CH}_3
\end{array}
\]

**Purpose:**

Metal dopants are used for isoelectronic temperature measurements in long scale length plasma experiments.
**Previous Work:**

Very low density PMP foams were largely developed at Oak Ridge; Naphthalene / Durene, foam densities as low as 5 mg/cc.

Joel Williams and Paul Apen at Los Alamos; densities <4 mg/cc.

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**Process**

Naphthalene 4 ml
Durene 6 ml
PMP 40 mg
Galvanoxyli 4 mg

180°C

→

Gel formation
cool to 80°C

→

Solidify

80°C

5°C

Machine and then evaporate the naphthalene and durene.
<table>
<thead>
<tr>
<th>Sample (mg/cc)</th>
<th>Ti wt%</th>
<th>Cr wt%</th>
<th>Ti / Cr</th>
<th>Cl wt%</th>
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</thead>
<tbody>
<tr>
<td>24</td>
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<td>0.7</td>
<td>0.4</td>
<td>1.75</td>
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<tr>
<td>3.6</td>
<td>0.29</td>
<td>0.14</td>
<td>2.07</td>
<td></td>
</tr>
</tbody>
</table>

**Metal Dopants:**

Add soluble form of metal at the beginning of the process.

- 0.1 M solution of TiCl₃ in methylene chloride
- 0.1 M solution of CrCl₃ in tetrahydrofuran
- 0.1 M solution of Mn(OAc)₃·2H₂O in ethanol

Very low density foams can be doped from 0.2 to 1 wt% (about 0.02 to 0.1 atom%).

The process becomes more difficult when the wt% approaches 1%.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Ti wt%</th>
<th>Mn wt%</th>
<th>Cl wt%</th>
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</thead>
<tbody>
<tr>
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<tr>
<td>30</td>
<td>0.4</td>
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</tbody>
</table>
Chlorine Loss:

Chlorine content is lower than expected.

GC/MS studies indicate that reactions with the solvent may be occurring.
Capabilities:

Low density (<4 mg/cc) CH foams can be produced with:

0.2 to 1 wt% Ti, Cr, or Mn

Other elements provided organic soluble compounds are available
THE STRENGTH AND OTHER INFORMATION ON HITACHI L-100 POLYIMIDE FILMS*

Edmund J. Hsieh, Ricke D. Behymer, Jorge J. Sanchez,
Marita R. Spragge, and Russell J. Wallace
University of California
Lawrence Livermore National Laboratory
F.O. Box 808, Livermore, CA 94551

With the advent of gas hohlraums, the search is on to find the best materials for the windows of the laser entrance holes and the diagnostic holes. The basic requirements of the windows are to hold the required gas pressures and to have minimum interference with the laser beams and the X-rays from the experiment. Thus materials with high tensile strength and low-Z are the best candidates. The Hitachi L-100 Polyimide films may be the strongest CH based polymer films suitable for the window applications. We experimented with the L-100 films of their burst strength on hohlraums and test tubes. The calculated tensile strength compared favorably with the published value. The same calculation may be used to estimate the minimum film thickness required for a given application. The strength calculations and other information of the L-100 films will be presented.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
The Strength and Other Information on Hitachi L-100 Polyimide Films

Presented to:
10th Target Fabrication Specialists' Meeting
Taos, NM
February 6-9, 1995

Edmund J. Hsieh
Ricke D. Behymer
Jorge J. Sanchez
Marita R. Spragge
Russell J. Wallace

University of California
Lawrence Livermore National Laboratory
BASIC REQUIREMENTS OF A WINDOW FOR GAS HOHLRAUMS

- WITHSTANDS 1 ATM OF PRESSURE
- PRESENTS MINIMUM OBSTRUCTION TO EXP.
  - LOW Z (POLYMERS)
  - LOW MASS (HIGH STRENGTH)

STRENGTH COMPARISON CHART FOR COMMON POLYMERS

<table>
<thead>
<tr>
<th>POLYMER</th>
<th>TENSILE STRENGTH</th>
<th>ATOMIC %</th>
<th>DENSITY</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
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<td>POLYSTYRENE</td>
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<td>MYLAR</td>
<td>1000</td>
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<tr>
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<td>50</td>
</tr>
<tr>
<td>-C</td>
<td>800</td>
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</tr>
<tr>
<td>-D</td>
<td>800</td>
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<td>340</td>
<td>34</td>
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<td>KEVLAR</td>
<td>27600</td>
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<tr>
<td>POLYIMIDE, generic</td>
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<td>56</td>
<td>26</td>
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<td>PIQ-13</td>
<td>1200</td>
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<tr>
<td>PIQ-L100</td>
<td>3200</td>
<td></td>
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</tr>
<tr>
<td>PIQ-L200</td>
<td>3100</td>
<td></td>
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</tbody>
</table>
STATUS OF OUR POLYIMIDE PROJECT

- PRIOR ART
- FILMS OF 2000Å TO 9000Å
- EXPERIMENTATION WITH EPOXIES AND ADHESIVES
- DELIVERY OF \( \approx 100 \) GAS TARGETS

GLUES WE HAVE TRIED & THEIR PROPERTIES

<table>
<thead>
<tr>
<th>EPOXIS</th>
<th>LIGHT CURED ACRYLIC ADHESIVES</th>
</tr>
</thead>
<tbody>
<tr>
<td>HEXCEL EPOLITE</td>
<td>UV</td>
</tr>
<tr>
<td>2409 RESIN</td>
<td>Dymax 628-Gel</td>
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<tr>
<td>2180 HARDENER</td>
<td></td>
</tr>
<tr>
<td>HARDMAN</td>
<td>VISIBLE</td>
</tr>
<tr>
<td>RED 04001</td>
<td>Dymax 4-20272</td>
</tr>
<tr>
<td>BLUE 04005</td>
<td>4-20260</td>
</tr>
<tr>
<td>BLACK 04006</td>
<td>3-20262</td>
</tr>
<tr>
<td>GREEB 04004</td>
<td>MICROLITE TECH A-17-M3</td>
</tr>
<tr>
<td>NOSAG</td>
<td>NORLAND OPTICAL #72</td>
</tr>
</tbody>
</table>
SOME OF THE QUESTIONS WE ASKED OURSELVES

- IS THE FILM WE MADE AS STRONG AS IT SHOULD BE?

- CAN WE DEFINE A TESTING CONDITION WHICH WILL QUALIFY THE FILMS TO BE USED UNDER ANOTHER SITUATION?

- BASED ON THE KNOWN TENSILE STRENGTH, CAN WE ESTIMATE THE MINIMUM FILM THICKNESS FOR A GIVEN WINDOW OPENING AND PRESSURE?

Cross section diagram of a pressurized LEH window

- $\Delta h$ – bulge height
- $a-c$ – window diameter
- $R$ – radius of curvature
- $P$ – pressure on window
STRENGTH OF OUR POLYIMIDE FILMS

\[ \sigma = \frac{PR}{2t} \]

where \( \sigma \), the tensile stress in film
\( P \), the pressure differential
\( R \), the radius of curvature
\( t \), the film thickness

\[ \tan^{-1} \alpha = \frac{\Delta h}{0.5ac}, \text{ thus } \theta = 92' \]

\[ \sin 0.5 \theta = \frac{0.5ac}{R}, \text{ thus } R = 830u \]

\( \sigma = \frac{PR}{2t} = 2980 \text{ Atm} \)

EQUIVALENT TEST CONDITION FOR FILMS

- KNOWN: \( \sigma_1 (P_1, D_1, t_1) \)

- TO FIND \( P_2 \) SUCH THAT

\( \sigma_2 (P_2, D_2, t_1) = \sigma_1 \)
EQUIVALENT TEST CONDITION FOR FILMS (cont.)

Exp. data

\[ \Delta h = 150 \text{u} \]
\[ D_1 = 1200 \text{u} \]
\[ t_1 = 0.35 \text{u} \]
\[ P_1 = 1.32 \text{ Atm} \]

\[ \angle 1 = 15^\circ \]
\[ \angle \theta = 60^\circ \]
\[ R_1 = 1200 \text{u} \]
\[ \sigma_1 = 2260 \text{ Atm} \]

There are two variables. We need to determine from the exp. R Vs P curve the value of \( P_2 \) such that,

\[ P_2 R_2 = 2260 \text{ Atm} \times (0.7 \text{u}) \]

\[ P_2 = 0.33 \text{ Atm} \]

Testing films on a 2500u opening at 0.33 Atm is equivalent to that of a 1200u opening at 1.32 Atm.
THINNEST FILM FOR A GIVEN OPENING

• KNOWN: % ELONGATION OF A FILM BEFORE BREAKAGE.

• TO FIND MINIMUM FILM THICKNESS BASED ON THE ASSUMPTION THAT SIMILAR FILMS WILL BREAK WHEN STRETCHED TO REACH THAT % ELONGATION

THINNEST FILM FOR A GIVEN OPENING (cont.)

CALCULATION OF % ELONGATION AT BREAKAGE.

\[ \text{Exp. data} \]
\[ D = 1200 \text{u} \]
\[ \sigma = 3200 \text{Atm} \]
\[ \Delta h = 250 \text{u} \]
\[ R = 833 \text{u} \]

\[ \text{cord } ac = 1200 \text{u} \]

\[ \text{arc } ac = \frac{\pi R\theta}{180^\circ} = 1338 \text{u} \]

\[ \frac{1338 - 1200}{1200} = 11.5\% \]
THINNEST FILM FOR A GIVEN OPENING (cont.)

\[ \text{cord ac} = 1600u = 2R \sin \theta/2 \quad (1) \]

\[ \text{arc ac} = 1600u \times 1.115 = \pi R \theta / 180^\circ \quad (2) \]

\[ \theta \quad \text{and} \quad \theta \quad \text{are unknowns.} \]

\[ \theta \quad = 128; \quad \theta \quad = 90^\circ, \quad R \quad = 1140u \]

\[ \frac{\sin \theta/2}{\text{Exp. data}} \]

\[ D = 1600u \]

\[ \sigma = 3200 \text{Atm} \]

\[ P = 1 \text{ Atm} \]

\[ t = ? \]

\[ t = \frac{P \times R}{2 \times \sigma} = 0.179u \]

STATUS OF OUR POLYIMIDE PROJECT

- FILMS OF 2000Å TO 9000Å (Hitachi L-100)
  * Strength
  * Testing
  * Minimum thickness

- EXPERIMENTATION WITH EPOXIES AND ADHESIVES
  * Light cured adhesives have a place in gas target assembly

- DELIVERY OF \( \approx \) 100 GAS TARGETS
ULTRAHIGH VACUUM FOCUSSED ION BEAM MACHINING OF COMPLEX MICRON- AND SUBMICRON SCALE ICF STRUCTURES

Bruce C. Lamartine
Los Alamos National Laboratory
P.O. Box 1663 MS-E549, Los Alamos, New Mexico 87545

The efficiency of small feature machining by focussed ion beam sputter milling has been previously limited by: (1) a vacuum-related limit on the aspect ratio of structures such as holes and grooves due to redeposition of previously sputtered material by ambient scattering and (2) a lack of sophistication in beam control hardware and software.

In the 70-80 pico Torr vacuum range, we have consistently milled holes of 1-3 microns diameter with aspect ratios (length/diameter) approaching 45 in copper and titanium without the use of adgas enhancement. At lower feature aspect ratios, we have used our beam control software to mill such complex structures as quadric and trigonometric surfaces in metals and silicon.

We will discuss specific examples of planar and spherical deformation structure fabrication by this technique and point out some of its remaining limitations.
During the production of window molds, the effect of micromachining variables, such as tool radius, tool rake, final cut depth, and final cut speed, on the surface finish of copper was determined. Machining was carried out by single-point diamond turning in a Precitech Optimum 2000 lathe, and surface finishes were determined qualitatively by optical microscopy and quantitatively by atomic force microscopy.
THE EFFECT OF MICROMACHINING VARIABLES ON THE SURFACE FINISH OF COPPER

by

J.L. KAAE and D.R. WALL

Presented to
Target Fabrication Specialists Meeting
Taos, New Mexico

FEBRUARY 6–10, 1995
APPEARANCE OF A SURFACE CUT WITH A DIAMOND TOOL
A SIMPLE MODEL OF A SURFACE CUT WITH A CIRCULAR TIP

\[ v = \text{cross head speed} \]
\[ w = \text{spindle rotation speed} \]

Then \( f = R - \left[ R^2 - \frac{v^2}{4w^2} \right]^{1/2} \) for \( R > \frac{v}{2w} \)

THEORETICAL EFFECT OF TOOL RADIUS, CROSS HEAD SPEED AND SPINDLE ROTATION ON SURFACE FINISH
1. Mat'1 O.F.H.C.
2. Quantity 50
3. Radius of 0.004 to 0.010 in. adequate
ROUGHNESS ANALYSIS OF A SURFACE CUT WITH
$R = 0.0250 \text{ mm AND } v/w = 6.25 \times 10^{-3} \text{ mm}$

Peak Surface Area Summit Zero Crossing Stopband Execute Clear

Roughness Analysis

Image Statistics

$Z_{\text{range}}$ 701.85 nm
Mean 0.001 nm
Rms (Rq) 121.94 nm
Mean roughness (Ra) 101.90 nm
Max height (Rmax) 701.93 nm

Box Statistics

$Z_{\text{range}}$ 356.33 nm
Mean -27.046 nm
Rms (Rq) 87.020 nm
Mean roughness (Ra) 62.375 nm
Max height (Rmax) 405.80 nm
Box x dimension 11.586 nm
Box y dimension 13.215 nm

GENERAL ATOMICS

ROUGHNESS ANALYSIS OF A SURFACE CUT WITH
$R = 0.760 \text{ mm AND } v/w = 3.75 \times 10^{-4} \text{ mm}$

Peak Surface Area Summit Zero Crossing Stopband Execute Clear

Roughness Analysis

Image Statistics

$Z_{\text{range}}$ 68.279 nm
Mean 0.00006 nm
Rms (Rq) 11.719 nm
Mean roughness (Ra) 9.041 nm
Max height (Rmax) 68.642 nm

Box Statistics

$Z_{\text{range}}$ 14.292 nm
Mean -3.074 nm
Rms (Rq) 2.846 nm
Mean roughness (Ra) 2.353 nm
Max height (Rmax) 15.069 nm
Box x dimension 11.405 nm
Box y dimension 13.396 nm

GENERAL ATOMICS
COMPARISON OF OBSERVED RMS ROUGHNESS VALUES WITH THEORETICAL PEAK TO VALLEY VALUES

Tool Radius 0.76 mm
Cut Depth 0.002 mm

- RMS of a 100 μm by 100 μm area
- RMS of smoothest 10 μm by 10 μm area

Tool Radius 0.025 mm
Cut Depth 0.002 mm

- RMS of a 100 μm by 100 μm area
- RMS of smoothest 10 μm by 10 μm area

Surface Finish (Å)

v/w (mm x 10^-4)
## EFFECT OF CUT DEPTH ON SURFACE FINISH

### TOOL RADIUS 0.025 mm

<table>
<thead>
<tr>
<th>( v/w ) (mm)</th>
<th>Cut Depth 0.002 mm</th>
<th>Cut Depth 0.010 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>( 3.75 \times 10^{-4} )</td>
<td>249 RMS</td>
<td>258 RMS</td>
</tr>
<tr>
<td>( 1.25 \times 10^{-3} )</td>
<td>212 RMS</td>
<td>390 RMS</td>
</tr>
</tbody>
</table>

### TOOL RADIUS 0.760 mm

<table>
<thead>
<tr>
<th>( v/w ) (mm)</th>
<th>Cut Depth 0.002 mm</th>
<th>Cut Depth 0.010 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>( 3.75 \times 10^{-4} )</td>
<td>94 RMS</td>
<td>117 RMS</td>
</tr>
<tr>
<td>( 1.25 \times 10^{-3} )</td>
<td>77 RMS</td>
<td>109 RMS</td>
</tr>
</tbody>
</table>

## SUMMARY

- Theoretically, the surface finish should depend only on the tool radius and the ratio of cross head speed to spindle rotation speed.

- Observed surface finishes do not correspond to theoretically predicted finishes except at high cross head speeds.

- The surface finish improves with increasing tool radius.

- For a given tool radius the surface finish does not vary strongly with the ratio of cross head speed to spindle rotation speed or with the cut depth.
CURRENT DEVELOPMENTS, PROBLEMS AND MINOR SUCCESSES
IN TARGET FABRICATION AT LOS ALAMOS

Peter L. Gobby, Michael A. Mitchell, Joyce E. Moore, Veronica M. Gomez,
Harry Bush, Jr., and Gerald Rivera
Los Alamos National Laboratory
P.O. Box 1663 MS E549, Los Alamos, NM 87545

Many aspects of target fabrication and assembly require developments, but perhaps do
not warrant dedicated presentations or papers. This presentation attempts to describe a
number of recent problems and developments that fall in this category. Topics to be
discussed include: 1) 1 μm wall parylene gas-filled spheres formed by coating the
inside of thicker walled shells, 2) improved fabrication techniques for multi-layered
cylinder targets using co-polymerization, and 3) acrylate foam-mitigated Rayleigh-
Taylor samples for 1-dimensional experiments. All the above represent either recent or
current developments.
CURRENT DEVELOPMENTS
at
LOS ALAMOS

P. L. Gobby, M. A. Mitchell, J. E. Moore,
V. M. Gomez, H. Bush Jr., and G. Rivera

LOS ALAMOS NATIONAL LABORATORY
PARYLENE CONFORMALLY COATS INSIDE OF NICKEL SPHERE

1 MM DIAMETER, 0.7 MICRON WALL

11.3 p.s.i. (1 Los Alamos Atmosphere)
Spherical hohlraum after backmachining

700 micron I.D.
30 micron wall

5 micron
MYLAR AND POLYCARBONATE ARE SWOLLEN BY SUPERCRITICAL CO2

FOAM MITIGATED DIRECT DRIVE

GOLD, 200 A

LASER

CHO, 50 microns
50 mg/cc

COPPER 50 microns
PARYLENE IS UNAFFECTED BY SUPERCRITICAL CO₂

PROBLEMS
PRECUSOR LEAKAGE WITH PARYLENE
CO₂ SWELLING OF EPOXY, PS, PC, PET

SOLUTION?

FORMVAR 800 Å

COPPER WASHER

THEN PARYLENE COAT
We have developed millimeter-scale targets for high-energy density physics experiments conducted on the Nova laser. We will describe two specific targets with unique features: one has a silica aerogel tracer disk, the other an aluminum-resin witness strip. These features facilitate radiographic imaging of the phenomena of interest. We will discuss target construction and characterization, and present a sampling of the experimental data.
Miniature Targets for High-Energy Density Experiments on Nova

Tenth Target Fabrication Specialists Meeting
February 6-9, 1995

Hedley Louis, Tony Demirli
K. Budil, P. Miller, T. Peyser, N. Woolsey
Lawrence Livermore National Laboratory
Topics to be presented

- Richtmyer-Meshkov instability at a high-to-low density interface

- 2 Nova target designs
  Construction & inspection
  Material synthesis & characterization

- A sampling of experimental data

Instability studies

**What:** To explore the physics of a shock-induced fluid instability (Richtmyer-Meshkov) which occurs when a shock wave passes over a density discontinuity between two materials. Roughness or other perturbations at the density interface grow in size after the passage of the shock, eventually leading to turbulence and mixing between the materials.

**Why:** The process is important in ICF target implosions and defense science.

**How:** Nova 10-beam, indirect drive experiments
1D linear sawtooth pillbox targets consist of several intricate parts:

- Tophat ablator, laminated CH plastics
- Hohlraum coupling, gold
- Hydro tamper, beryllium
- Tracer disk, silica aerogel
- Payload, carbonized organic aerogel
Close-up view of a mix target mounted to a hohlraum reveals some requisite features

Small tophat-to-hohlraum radial clearance and gold coupling-to-hohlraum flat mounting minimize radiation leakage.

Re-entry tophat ablator yields a planar wave shock front at the high-to-low density interface

High-density component, $\rho = 1.22/1.05 \text{ g/cm}^3$

**Material synthesis & characterization**

**Target construction & Inspection**
* Lamination-$(\text{C}_5\text{H}_5\text{NO}_2)_n$ cyano-acrylate adhesive
* Preliminary machining-CH disk to finished thickness
* Sawtooth forming in sequential steps using a diamond tool-nearly linear 60-degree, close-coupled V grooves of amplitude 5, 10, or 21.5 $\mu$m
* Final machining on a diamond turning lathe
* Inspection-SEM cross-section
Allied Signal has synthesized high quality polystyrene rods at 1, 2, and 3 at% bromine.

**Process:**

1. Mix 4-bromostyrene ($\text{C}_8\text{H}_7\text{Br}$) monomer and styrene ($\text{C}_8\text{H}_8$) monomer; add catalyst
2. Pour into a glass tube having one closed end
3. Evacuate; then seal other end of tube
4. Heat in 75°C furnace; polymerize into a 3-mm-diameter rod

**Advantage:** homogeneous co-polymer

**Characterization:**

Bromine content (20.5 wt %) by ignition of a BrCH sample in an oxygen flask, then titration with $\text{Ag}^+$ (2 at% Br = 19.8 % by weight)

Gravimetric density 1.22 g/cm³ (3 mm dia by 20 mm)

Contact Mike Smith at Allied Signal for further information on the process.

SEM cross-section shows a typical sawtooth having sharply-defined apex and roots.

Encapsulated specimen in Al$_2$O$_3$ resin, sectioned, then polished for viewing under an SEM.

3,000x magnification of 60-degree V grooves of amplitude 10 µm.
Silica aerogel tracer disk facilitates radiographic imaging of the phenomena of interest

0.3 dia by 0.125 mm, \( \rho = 0.1 \text{ g/cm}^3 \)

** Casting process **

** Characterization **

Gravimetric density of a witness sample: 0.10 g/cm\(^3\) (1-mm-dia by 25 mm)

Inspection of tracer disks under a microscope

Pre-formed silica aerogel disks are the smallest ever made at LLNL

0.3 mm dia. holes machined in 0.025 mm thick foil

Contact Larry Hrubesh at LLNL for further Information on the process
Carbonized organic aerogel payload is the low density component of a mix target

0.5 dia x 1.9 mm, 0.3 dia x 0.125 mm c'bore, \( p = 0.1 \text{ g/cm}^3 \)

**Material synthesis & characterization**

**Target construction & inspection**
* Diameter & length machined on a diamond turning lathe
* Counter bore machined with a modified 40-degree, zero radius diamond tool
* Dimensional inspection under an optical microscope
* Surface roughness by SEM
Hydro tamper & hohlraum coupling

The tamper also serves to hold the payload in place

* Beryllium hydro tamper: 0.5 ID x 0.7 OD x 2.025 mm
  ID machined with a spade drill;
  roundness 0.002 mm
  OD machined with a CBN tool

* Gold coupling machined on a diamond turning lathe

* C43H49O8 epoxy fillet joint

Nova hydrodynamics SiO2 compressible high opacity tracer layer experiment: schematic
Early time X-Ray streak camera image of silicon aerogel tracer layer target

- 3 ns square Ti backlighter (4.7 keV) t=5.0 to 8.0 ns
- Shock wave compresses tracer layer from initial 125 µm to =15 µm
- Compressed tracer layer displaced by shock wave

![Diagram of shock wave approaching and compressed tracer layer trajectory]

Later time X-Ray streak camera image of silicon aerogel tracer layer target

- 3 ns square Ti backlighter (4.7 keV) t=7.5 to 10.5 ns
- Compressed tracer layer appears to follow shock trajectory
- Tracer layer expansion at 10.5 ns minimal (=25-30 µm)

![Diagram of compressed tracer layer boundary and spatial fiducials]
Nova hydrodynamics Al tracer for shock tube boundary layer experiment: schematic

Aluminum tracer layer permits study of shock tube wall expansion (boundary layer)

Aluminum resin stripe highlights the edge effect of a BrCH/CH target

Material: aluminum-resin, ~45 wt% aluminum powder by chemical analysis of a sample

Channel: constant cross-section 0.050 wide by 0.020 mm, machined with a fly-cutter

Dimensional inspection: measured under an optical microscope, both ends
X-Ray framing camera image of Al tracer layer and shock tube wall expansion

- Hydrodynamically stable target (small density difference between payload and ablator)
- Sc backlighter (4.2 keV) t=15 ns
- Observed radial displacement of tracer layer due to shock

- Three regions in image:
  (1) compressed high opacity BrPS
  (2) compressed low opacity PS
  (3) uncompressed PS (ahead of shock wave)

In summary

We have described in detail the construction and characterization of an unique target to study mix, and presented a sampling of experimental data gathered on Nova
This paper describes gas filled hohlraums as built and fielded by LANL for the past NOVA Laser campaign and attempts to describe the future.

Target designs described are JCFLG the first gas filled hohlraum, and the SYMGAS the first gas filled symmetry hohlraum.

The gas handling systems for gas filled hohlraums are an important part of the experiments. The Target Test Stand and Gas Fill System, NOVA Target Pylon Integration, CAD 3D Model of NOVA Target Pylon, Full Scale Target Test Stand and Gas Fill System will be described.

High Pressure Gas Handling Systems will play an increasingly important roll in the drive to NIF like conditions. We have designed and built an \textit{in situ} high pressure gas fill system. We have designed a hohlraum to handle increased pressure and tested this new design to the required pressure.

There is a future for higher density gas hohlraums. Potential design variations for hohlraums can be speculated. One interesting approach to higher gas densities is by use of nitrogen cryogenics. This approach would allow experimental conditions to be reached that can not be reached today due to strengths of low mass materials.
Gas Filled Hohlraums

by

Mike A. Salazar, Larry Foreman, Pete Gobby, Gary Stone*, Harry Bush, Veronica M. Gomez, Joyce E. Moore

Los Alamos National Laboratory
University of California,
Department of Energy, U.S.A.

*Lawrence Livermore National Laboratory
The first targets were fielded with a gas holding reservoir. A one atmosphere gas load was the desired pressure. Various gas combinations were used. A stainless steel fill tube was bonded to the Au hohlraum with a polyurethane adhesive. A silicon nitride window was also bonded to the case with the same adhesive. These windows were supplied by Dino Ciarlo of LLNL. These windows are generally .25 microns thick with a frame that varies in thickness and shape.

Many of these targets incorporated various cut outs for diagnostics covered with thin films of various types. These diagnostic windows complicate the fabrication of gas tight targets greatly.

Thin wall Au targets of this general design with a thick epoxy layer were also fielded. These targets are designed to diagnose laser pointing.
Target Test Stand and Gas Reservoir Fill System

The fill system was designed to enable evacuating the target without putting an external pressure on the Silicon Nitride windows which rupture when stressed to the interior of the target. The Gas system consisted of a target base, small reservoir, valve, fill tube and target.

The target was mounted on a plate inside of a plastic jar. The valve mounted on the target system is open and a tube leading to a manifold connected. Air from inside the jar is removed, then air from the inside of the target is evacuated. The fill gas is introduced to the inside of the target. The jar is brought back to atmospheric pressure. The jar is removed and the target valve closed. A system that evacuates the air from the outside of the target and then the inside means that the target never experiences a force to the interior. This system also requires only one tube for evacuating and filling the target. This method simplifies construction and conserves gas. The gas mixing and fill system was designed and built by LLNL for LLNL gas experiments. We were able to use this system to handle gas for our test and fill stand.
2nd Generation Target Test Stand

Before the first targets were fielded a second generation design gas system was in progress. This system was designed in 3D CAD. This second generation system incorporated a pressure transducer that had to attach to the pylon and not interfere with the wall of the antechamber. The NOVA target pylon was modeled and the sub systems were designed to fit it and clear the antechamber of the NOVA laser target insertion mechanism. 3D modeling enabled a fit test on the CAD computer at a time when there was no opportunity to correct for errors in the design on site.

This second test fill stand enabled checking targets for fit, leaks and enables filling the targets with gas. This fill system worked well. Three more units were built to test and fill both LANL and LLNL target experiments. It is my understanding that this system is also being used to fill French experiments.
• These targets designed by Norm Delameter have a micro sphere filled with Argon and Helium gas at various mixes and pressures suspended in the center of the gas filled hohlraum.
• This target type is a complex construction when the hohlraum case does not hold gas.
• Making this target gas tight is difficult, time consuming, and requires great attention to detail.
• This type of target has been constructed with a variety of window types Silicon Nitride, Mylar, and Polyimide films.
High Pressure Gas Handling System

In Situ Fill System

- This third generation gas handling system has been designed with CAD and built to function in the NOVA system.
- Targets are very fragile things.
- Targets filled to higher pressure using the older methods would have to withstand these higher pressures for several hours.
- After alignment, diagnostic and other considerations have been met and within a few minutes of shot time this system can fill targets in situ to a pressure.
- This system can also do sub atmospheric fills should that become desirable.
- This third generation fill system is comprised of a manual valve, two small electrically actuated valves, two very small pressure transducers, reservoir and target.
- The integration of this third fill system with NOVA is being handled in cooperation with LLNL.
- We will be able to control and monitor filling operations from the NOVA control room.
- This capability extends the range of operations for gas targets.
Future High Density Gas Fill Systems

• The need for higher pressures may not be the only way to go.
• The reason we are going to higher pressures is really to achieve higher gas density.
• Higher density gas by use of liquid nitrogen cryogenics is possible.
• Their seems to be a way of incorporating cryogenics into the target pylon.
• We are looking at this possibility as the fourth generation fill system for targets at NOVA.

Conclusion

• Co-operation between National Laboratories is essential to achieving National Fusion Ignition goals.
• Computer Aided Design and Electronic Communication are a fact of life and provide a quick method of accurate communication of technical information world wide.
• We have been able to manufacture gas tight laser target holraums of various designs.
• More variations are possible.
• We will continue to build and field targets of advance design.
• We have designed and built various gas handling systems successfully.
• We will continue to expand this capability.
• Higher densities are the future.
Gas filled targets were required to study large scale plasma target conditions similar to the NIF point design - $1.0 \times 10^{21} \text{ electrons-cm}^2 / 3 \text{ keV}$. The specifications and designs for the target from the Theoretical and Experimental Groups were translated into targets for Nova. The fabrication of a small volume (~10 mm$^3$) gas cells, with thin low Z windows, capable of holding >1 ATM was required. A gas manifold with a pressure transducer and that operated inside the Nova Target chamber vacuum was required and fabricated. Gas handling hardware for mixing and target filling will be described. Testing procedures for various target materials components will be described along with the material choices for various target components. A description of the fabrication process from target specification to delivery to Nova will be discussed.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Fabrication and Testing of Gas Filled Targets for Large Scale Plasma Experiments on Nova

Gary F. Stone, Craig J. Rivers
Marita Spragge, Russell J. Wallace

Presented to:
Tenth Target Fabrication Specialists Meeting
Taos, New Mexico
February 6 - 9, 1995
Gas Cell Target Fabrication - Major Considerations

- Target Specification and Design
- Target material selection and window fabrication
- Component micro-machining
- Gas specification, mixing and analysis
- Gas mixing system design and fabrication
- Target manifold design and assembly
- Target pressure/vacuum testing hardware
- Target fabrication and final assembly
- Target filling system and filling procedures
- Target documentation and reporting

Gas Cell Target Fabrication - NIF Point Design

- NIF point design for closed geometry targets
- Gas filled target to simulate target conditions
- Plasma at ~ 3 kV temperature - $10^{21} \text{ cm}^{-3}$ electron density

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Gas Cell Target Fabrication - Target CAD Drawing

- CAD drawing - Scaled in 3D for interference checks
- Drawings assist in machining and metrology - QA/QC
- Drawings allow physicist to verify clear diagnostic LOS
- Micromachining identical to standard hohlraums

![CAD Drawing Image]

Gas Cell Target Fabrication - Target Types

- Open and closed geometry - diagnostic views - LOS
- Open geometry - nearly spherical CH balloons
- Closed geometry - hohlraum based targets

![Target Types Images]
Gas Cell Target Fabrication - Gas Specification

- Simulate $10^{21}$ electrons cm$^3$ with a gas at 1 ATM
- Neopentane - C$_5$H$_{12}$ - produce 1.07 E21 if fully ionized
- Ar & CClF$_3$ added for spectroscopic measurements
- Lower densities used CO$_2$, CH$_4$, C$_2$H$_6$ as base gas
- Deuterated neopentane permitted neutron diagnostics
- Typical mixture was 1% Ar, 1% CClF$_3$ and 98% base gas
- Mass spec used for gas analysis - 0.01% resolution
- Odd gas requests - CN, TiCl$_4$, dopants, C$_5$T$_{12}$, Xe+C$_3$H$_6$
  \[ C_3H_8 + C_5D_{12} + Ar + CClF_3 \]

Gas Cell Target Fabrication - Gas Mixing system

- Original system used for gas mixing & target filling
- Dedicated system placed in fume hood - flammable gasses

Main gas mixing manifold valves
50% Ar / 50% CClF3 gas cylinder
Auxiliary gas valve manifold
MKS Baratron pressure readout
Gas Cell Target Fabrication - Target manifold

- Gas tight assembly with pressure transducer & valves
- Capable of multiple shots in vacuum chamber - re-usable

Gas Cell Target Fabrication - Testing system

- Completely independent from gas target filling system
- Capable of testing two gas targets at one time
Gas Cell Target Fabrication - Target Fabrication

- Open geometry formed on 0.4 mm washer - radial fill tubes
- Thin polyimide windows (0.35 - 0.90 μm) glued on flats
- GASBAG balloon formed by 25 - 35 PSI over pressure

- Closed geometry based on "standard" Nova hohlraums
- Gold coated copper mandrel micro-drilled and milled for holes
- HNO₃ etch leaving gold hohlraum remaining
- Fill tubes, patches, Witness plates attached gas tight
- Thin polyimide windows (0.20 - 0.65 μm) over holes, slots

- All targets leak checked at >17 PSI - prior to assembly
- Plastic fill tubes attached from target fill lines to manifold
- Final assembly re-tested for leaks prior to gas filling

Gas Cell Target Fabrication - Gas filling System

- Original system was flow through - inefficient / expensive
- Evacuation / backfill system developed by LANL was adopted

Target transducer power supply & voltage readout
Absolutely calibrated transducer readout
Mechanical manometer 0 - 200 Torr

Absolutely calibrated transducer
Gas filling manifold
Gas sample bottle - 1 liter
Gas Cell Target Fabrication - Target Documentation

- CAD drawings provided to shot physicist for verification
- Target information kept on VAX / ORACLE database
- Photographs during fabrication - metrology by Assembly
- Target window specifications - log file to shot physicist
- Gas analysis data sent to shot physicist in log file
- Target transducer calibration data in logbook - Unique number
- Calibration values for transducer sent over with target to Nova
- Target testing and filling procedures in check lists
- Corporate "memory" documented in reference manual

Gas Cell Target Fabrication - Conclusions

- NIF point design targets tested on Nova
- Open and closed geometry gas cell target fabricated
- Thin CH window technology adapted for gas tight targets
- Gas target density varied during experiments
- Spectroscopic gasses, foils and fibers added to targets
- 350+ gas cell targets fielded successfully on Nova
Posters
The Particle Injector for the Ballistic Furnace

S.M. Tolokonnikov, V.S. Bushuev, and A.I. Nikitenko
P.N. Lebedev Physical Institute, Russian Academy of Sciences
Moscow, Russia

For the manufacture of large (2 mm or more in diameter) microshells suitable for NIF experiments, the concept of a “ballistic furnace” was suggested by scientists at LPI. In this furnace the initial particles are propelled upwards into the heat zone along a ballistic trajectory. This approach allows one to sharply increase the time the particle spends in the hot zone of the furnace, as well as allowing microshell formation to occur near the trajectory apex when its velocity is small. The key unit of the “ballistic furnace” is the injection device that accurately propels the initial particles from below with a given speed and precisely controlled trajectory without any damage to the accelerated particle. An electromagnetic device that satisfies these needs is described and the results of its tests are presented. This system can be also be used for the accurate delivery of targets into the laser focus of a multi-beam ICF installation.
PRODUCTION OF MICROSPHERES FROM SOLID PLASTIC GRANULES

S.A. Startsev, A.A. Akunets, V.M. Dorogotovtsev, Yu.A. Merkuliev
P. N. Lebedev Physical Institute, Russian Academy of Sciences
Moscow, Russia

Rober Cook
Lawrence Livermore National Laboratory
Livermore, CA

Work at the P. N. Lebedev Physical Institute on hollow microsphere technology started more than twenty years ago, based on the commercial technology developed in our country, on the experience of our colleagues involved in chemical research, and on our knowledge of physics and hydrodynamics. Unlike the microencapsulation or solution drop tower techniques used for plastic microshell formation in the U.S. and elsewhere, we have developed a drop tower technology for producing hollow plastic shells from solid plastic granules infused with a volatile component, which serves as the blowing agent. Using these techniques we have been able to produce high quality plastic shells with diameters as large as 1.5 mm.

In this paper we will present the essential technological features of the process along with the elements of a theoretical model that explains the physics of the shell formation process. We will also present detailed characterization of polystyrene shells with diameters from 1.0 to 1.5 mm produced using these techniques.

Part of this work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Production of Hollow Microspheres from Plastic Solid Granules


Lebedev Physical Institute, Russian Academy of Sciences

*Lawrence Livermore National Laboratory.
Our first polystyrene shells for laser targets were manufactured in May - June 1974. A.I.Isakov and O.N.Kozhm showed the targets in Tokyo conference in the same year. Initial granules from deuterated polystyrene were prepared (emulsion method) by collaborators of Academician V.V.Korshak in the Moscow Chemistry-Technology Institute.

These activities were resulted by the review "Polymer Laser Shells" in vol.127 Trudy FIAN 1980.

In 1981 - 1984 in the process of works by theme "Storage of Hydrogen in Microballoons" we had developed method polyethylene-terephthalate shells manufacture (polymer base - maylay). It was possible to storage hydrogen for short time at room temperature and pressure about 100 atm in the shells. The strength of the shells increased 2 - 3 times and gas penetrability reduced by temperature reducing down to 80 K.

Polymer shells - targets were used in laser experiments not as often as glass targets. Because the glass targets allow to get more information in X-rays.

Fig. 1. Installation for emulsion polymerization of polystyrene
1. - monomer solution with catalyst and surface-active substance.
2. - autoclave.
3. - gas admission at pressure up to 12 atm.
4. - rod.
5. - circulation rate control motor.
6. - motor driver unit.
7. - pressure sensor.
8. - thermostat.
9. - thermostat driver unit.
10. - vessel for emulsion of water in styrene.
Inlet

Heating during hovering, melting

Gas isolation inside the drop, nucleation centre initiation (leader growth)

Gas expansion (cavity growth)

Cooling; compression and solidification

Yield outstage

Fig. 1. Physical processes performed in hollow microsphere fabrication technology.
1182 µm shell
1238 μm shell

1464 μm shell
1 mm plastic shells from LPI have roughness amplitude at a given wavelength similar to LLNL shells.

On a 500 µm LLNL shell a 50 µm wavelength is mode 10. The same wavelength on a 1 mm shell is mode 20.
Power spectra from recently prepared 1 to 1.5 mm shells.

Shells are rougher than LLNL shells at the same mode number.

Power spectra from recently prepared 1 to 1.5 mm shells.

Comparison at same wavelength
Mode number has been scaled by shell diameter.
1. THE RETRADIATION EQUATIONS FOR DESCRIPTION OF MICROSPHERE MOTION IN GAS

\[ m \ddot{Z} = m \dot{Z} \frac{dZ}{dz} = mg - Cd \rho_0 \frac{\dot{Z}^2}{2} \pi R_o^2; \text{ where } Re = \frac{2R_o \dot{Z}}{\nu} - \text{Reynolds number}, \]

\[ Cd = Cd(Re) - \text{experimental dependence of resistance coefficient}. \]

2. HEAT CONDUCTIVITY EQUATION AND BOUNDARY CONDITIONS FOR SPHERICALLY SYMMETRIC CASE

\[ \lambda \frac{\partial T}{\partial r} \bigg|_{r=r_o} = \alpha_g (T_g - T_{r_o}) + A \sigma (T_g^4 - T_{r_o}^4); \ \frac{\partial T}{\partial r} \bigg|_{r=r_i} = 0. \]

3. DIFFUSION EQUATION FOR GAS-FOAMER WITH BOUNDARY CONDITIONS

\[ \frac{\partial C}{\partial t} + r \frac{\partial C}{\partial r} = \frac{1}{r^2} \frac{\partial}{\partial r} \left( r^2 D \frac{\partial C}{\partial r} \right); \ C(R_o, t) = C_o; \ \frac{d \rho}{dt} = 4\pi r^2 D \frac{\partial C}{\partial r} \bigg|_{r=R_i}; \ C_i = \Gamma P_i. \]

\[ \Gamma(T) = \Gamma_c \left( \frac{T_c}{T} \right)^\gamma \]

\[ D(T) = D_0 \exp\left[-\frac{E_0}{kT}\right] \]

4. HYDRODYNAMICAL EQUATION, CONTINUITY EQUATION AND BOUNDARY CONDITIONS

\[ \rho \left( \dot{r}_i + \dot{r}_i \frac{\partial r}{\partial r} \right) = - \frac{\partial P}{\partial r}; \ \frac{\partial}{\partial r} (r^2 \dot{r}) = 0 \]

\[ P - 2\eta \frac{\partial \dot{r}}{\partial r} = P_i - \frac{2\sigma}{r_i} \quad \text{for } r(t) = r_i \]

\[ P - 2\eta \frac{\partial \dot{r}}{\partial r} = P_o + \frac{2\sigma}{r_o} \quad \text{for } r(t) = r_o \]

5. NUCLEATION EQUATION

\[ J = N_1 \sqrt{\frac{2\sigma}{\pi M}} \exp\left(-\frac{W}{kT}\right) \]

\[ W = \frac{16\pi^3 \sigma^3}{3(P - P_o)^2 \left(1 - \frac{V_i}{V_o}\right)^2} \]
PREPARATION OF PAMS BEADS AND SHELLS AND EFFECTS OF PLASMA COATING

S.A. Letts, E.M. Fearon, S.R. Buckley, M.D. Saculla, L.M. Allison, and R.C. Cook
University of California
Lawrence Livermore National Laboratory
P.O. Box 808, Livermore, CA 94551

To successfully develop the depolymerizing mandrel method for preparing microshells, techniques needed to be developed for preparation of smooth, spherical poly(alphamethyl styrene) (PAMS) mandrels. Initial experiments used hot water to soften the PAMS and allow surface tension to form the material into a sphere. When multiple beads were present in hot water, the particles aggregated. Use of PVA as a surfactant prevented aggregation and also improved the surface finish. Surface finish can be further improved by passing the beads through solvent vapor, solvating the outer layer, followed by drying in a drop tower.

Recent experiments have concentrated on two techniques for making hollow shells which have the advantage of having much less material to ultimately pyrolyze. Shells were first made by microencapsulation techniques. We also used a swollen frit method where pieces of PAMS swollen with toluene were blown into shells in a hot drop tower.

The beads or shells were next coated with plasma polymer. To deposit a uniform coating, the beads needed to be kept in constant random motion. Use of a slowly rolling pan resulted in poor coating uniformity and caused collapse of the coating during pyrolysis. During coating bubbles developed inside the PAMS bead if the coater power is high. This caused the bead to swell, fracturing the coating and leading to nonuniformity because of the off center bubble.

The thermal properties of plasma polymer were studied to determine whether shrinkage of the coating during the final pyrolysis phase could be reduced. We saw little difference between the plasma polymer collected from several different coaters running at different conditions. Shrinkage of coating was typically 10%.

• Work performed under the auspices of the U.S. Department of Energy Lawrence Livermore National Laboratory under contract number W-7405-ENG-48.
Preparation of PAMS Beads and Shells and Effects of Plasma Coating

S.A. Letts, E.M. Fearon, S.R. Buckley, M.D. Saculla, L.M. Allison, and R.C. Cook

Acknowledgements:
E.F. Lindsey, C.E. Moore, D. Schroen-Carey (WJSA), M. Whitman (UR/LLE)

Tenth Target Fabrication Specialists’ Meeting
Taos, New Mexico
February 6 – 9, 1995
A possible method for shell production uses thermally stable coating over a depolymerizable substrate. 

$\begin{align*}
&\text{CH}_3 \\
&(-\text{CH}_2-C-)_n \\
&\text{Heat} \\
&\text{280°C} \\
&\rightarrow \\
&\text{CH}_2=\text{C} \\
&\text{poly (α-methyl styrene)} \\
&\text{α-methyl styrene}
\end{align*}$

**Polymer shell**

**Polymer shell with metal layer**

Smooth spherical PaMS mandrels are made using thermal and vapor treatment.
Crushed PαMS beads are hot water treated to form spheres

Surface finish on the PAMS bead varies with treatment

PAMS melted in air has the smoothest surface finish—0.6 nm rms

PAMS treated in 88°C water/PVA solution—3.5 nm rms

PAMS melted in 88°C water—8.7 nm rms
The Sphere Mapper AFM characterizes sphericity and surface finish through the entire process.

A vapor treatment contactor was added to an existing drop tower to improve surface finish of PaMS.
Solvent vapor treating the hot water smoothed PAMS bead further improves its surface finish.

AFM surface profile hot water treated bead—rms 2.8 nm

Solvent vapor treated bead—rms 0.4 nm

Plasma polymerization conditions determine the uniformity of the final pyrolyzed shell.

Bouncing with electronic white noise

Drill
Solvent extract
Pyrolyze

Nonuniform
Pyrolyze
Collapsed

Rotating with mechanical transfer
The heat of plasma polymer coating can cause bubbles to grow within PAMS beads.

- Bubbles grow inside 11K Mw PAMS beads during plasma polymer coating.
- Imbalanced beads (caused by bubbles) lead to nonuniform coating thickness.
- Use of 19K Mw PAMS prevents bubble formation.

Rotation during pyrolysis results in shells with better sphericity.

Surface tension forces from liquid PAMS during pyrolysis may cause distortion of the shell.
Pyrolysis with rotation results in improved sphericity.

Shells up to 2 mm were made using a concentric needle droplet generator.
Microencapsulated shells show very good sphericity with a small number of surface defects.

Sphericity is maintained through pyrolysis using mandrel made by microencapsulation.

Pyrolyzed at 300°C, 2000 min.
Conclusions

Using a depolymerizing mandrel, shells were made from 200 μm to 4 mm diameter
Small beads made by grinding (200 μm – 1 mm)
Large beads made by melt sintering (4 mm)
Surface finish and sphericity are improved by treatment in hot water
  – Sphericity < 2 μm out of round
  – Roughness <10 nm RMS
Solvent vapor treatment in a drop tower improves surface finish to < 1 nm RMS
Optimal heating profile is rapidly ramp to 200°C, hold 30 minutes, then ramp at 0.2°/min to 300°C and hold for 2000 minutes (longer or shorter depending on size of shell)

Microencapsulated shell mandrels pyrolyze completely at 300°C in 1000 m
Solid bead mandrels pyrolyze completely at 300°C in 2000 m

290°C/2000 m

300°C/2000 m
The ICF program requires mandrels which contain 0.06 to 0.2 atom percent titanium for composite polymer capsules. We produced mandrels by the drop tower method using two different sources of titanium-doped polymer. One source is titanium-doped copolymer. Synthesis of the copolymer with predictable titanium content has proven to be difficult. Another source is a mixture of a low molecular weight, nonvolatile titanium compound with monodispersed polystyrene. Using titanium compound mixtures provides an inexpensive, quick, method of producing titanium-doped mandrels with any desired titanium content. We produced quality mandrels using both sources of polymer.

The mandrels produced by the two techniques are compared.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
WE CAN PRODUCE TITANIUM DOPED DROP TOWER SHELLS USING TWO DIFFERENT STARTING MATERIALS

- copolymer-Styrene titanium methacrylate
- mixture-Polystyrene/ low molecular weight titanium compound

SYNTHESIS OF STYRENE-TITANIUM METHACRYLATE COPOLYMER

\[
\begin{align*}
\text{CH}_3 & \quad \text{H}_2\text{C} = \text{C} \\
\text{H}_2\text{C} = \text{CH} & \quad \text{AIBN} \\
\text{O} = \text{C} - \text{O} - \text{CH}_2 - \text{CH}_2 - \text{O} - \text{Ti} - (\text{OC}_3\text{H}_7)_3 & \quad \text{C}_6\text{H}_6 \\
\text{Methacryloxy ethoxy titanium tri-isopropoxide} & \quad 60 \text{ C} \\
(\text{Gelest proprietary material}) & \\
\end{align*}
\]
CONTROL OF TITANIUM CONTENT OF THE COPOLYMER IS DIFFICULT

- Most Ti is lost from the methacrylate monomer during polymerization and workup.
- Increasing the ratio of Ti-methacrylate to styrene increases the amount of Ti in final polymer

![Graph showing the relationship between titanium content and monomer ratio]

*Atomic Percent oxygen (a/o O) calculated from NMR results

MIXTURE-SELECTING THE BEST ADDITIVE

- A number of additives were tried and rejected because:
  - The titanium compound evaporated during the blowing process
  - The additive precipitated the polymer from the solution
  - The compound wouldn't dissolve in methylene chloride
  - Seemed to 'gel' causing 'lumpy' shells

The best additive we found was:

\[
\text{titanium (IV)bis(ethylacetoacetato)-diisopropoxide (EtAcAc)}
\]

\[
\text{H}_3\text{C}\overset{\text{C}}{\text{CH}}\overset{\text{O}}{\text{O}}\overset{\text{C}_2\text{H}_5}
\]

\[
(\text{H}_3\text{C})_2\text{HC}\overset{\text{O}}{\text{O}}\overset{\text{Ti}}{\text{O}}\overset{\text{O}}{\text{CH(CH}_3)_2}
\]

\[
\text{H}_3\text{C}\overset{\text{C}}{\text{CH}}\overset{\text{O}}{\text{O}}\overset{\text{C}_2\text{H}_5}
\]

353
SELECTING THE BEST MIXING AND RUN CONDITIONS

NOTE: Moisture must be excluded at all steps of the process as water will precipitate the titanium from the solution.

Titanium Stock Solution
- A 10% solution of anhydrous methylene chloride and the titanium compound are mixed in a septa bottle.

Drop Tower Solution
- Dry polymer in a septa bottle in vacuum oven
- Transfer dry copolymer to septa bottle in dry box
- Add desired amount of anhydrous methylene chloride to septa bottle
- Add desired amount of titanium stock solution to septa bottle

Run Conditions
- Purge drop tower with nitrogen prior to run
- Each additive requires a slightly different temperature profile, within the drop tower, to produce desirable shells

WE HAVE MADE SHELLS FROM OTHER LOW MOLECULAR WEIGHT TITANIUM ADDITIVES

Titanium(IV) diisopropyl bis(2,4-pentanedionate) (0.113 a/o Ti)

Titanium tetrachloride tetrahydrofuran complex (0.093 a/o Ti)
THE ADDITIVE METHOD PROVIDES TITANIUM CONCENTRATION FLEXIBILITY AND QUICK TURN AROUND

- The desired amount of titanium can be added to the solution and end up in the shells
- Quick turn around time

We have controlled the titanium content in the shells over this range by controlling the titanium content in the solution.

EDXS DOES NOT SHOW SIGNIFICANT TITANIUM VARIABILITY WITHIN A SHELL

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LLNL SIMS DATA SHOWS NO SIGNIFICANT VARIATION IN TITANIUM CONTENT THROUGH SHELL WALL

![Graph showing count rates and relative counts for different isotopes over cycle number.](image)

SEM PHOTO AFTER SIMS ANALYSIS

![SEM photo of a sample after SIMS analysis.](image)
TWO METHODS USED FOR PRODUCING DROP TOWER TITANIUM DOPED SHELLS

Copolymer Shell | EtAcAc Shell

COPOLYMER METHOD

Advantage
- Shell wall thickness more uniform

Disadvantage
- High atomic percent Oxygen
- Polymer production requires at least three weeks of lead time
- Titanium content not easily varied nor controlled

ADDITIVE METHOD

Advantage
- Titanium content can be easily and precisely varied
- Quick turn around time

Disadvantage
- Wall thickness not as uniform
- More difficult to blow shells
MICROHETEROGENEOUS TARGETS - A NEW CHALLENGE
IN TARGET TECHNOLOGY, PLASMA PHYSICS,
AND LASER INTERACTION WITH MATTER

N.G. Borisenko, A.I. Gromov, and Yu.A. Merkuliev
P. N. Lebedev Physical Institute, Russian Academy of Sciences
Moscow, Russia

Targets with microheterogeneous structure, arising either unavoidably during the fabrication process or purposely incorporated, are discussed with respect to technology problems and plasma applications. The variety of microheterogeneous materials for laser targets ranges widely in both composition and density. We concentrate on certain types of polymer foams, 3-D nettings and plastic or glass with an quasi-lattice of metal clusters inside (3-D also). Uniform low-density metal layers made of snow-like ultradispersed powders on the surface of glass shells are also demonstrated.

We have used the microheterogenous structures for targets in ICF experiments and propose them for fundamental plasma research with existing drivers. We present microheterogenous or 3-D structured targets made a) for Raleigh-Taylor instability studies, b) for driver energy redistribution (equalization) over the target surface, c) as undercritical targets, d) for plasma self-organization, e) for regularly induced self-generated magnetic fields in ICF plasmas, and f) for highly non-ideal plasma studies.
POLYVINYL ALCOHOL (PVA) COATING PROCESS AT GENERAL ATOMICS

Jane B. Gibson, Wesley A. Baugh, Lloyd C. Brown, David O. Husband, Martin L. Hoppe, David A. Steinman, General Atomics, San Diego, CA 92186-9748

The current design of composite polymer capsules consists of polystyrene shells coated with PVA and a final glow discharge polymerization (GDP) coating. The PVA layer functions as a gas impermeable barrier to retain the fuel and diagnostic gases. Details of the PVA coating process now in operation at General Atomics will be described.

Historically, the PVA coating process has been of low yield. Recent modifications to our PVA coating process have resulted in increased yields. The major defects in PVA-coated shells were 'strings' of shells stuck together during the coating process and debris. We markedly reduced strings when we stopped evacuating the shells before coating with PVA. Previously this step was necessary because we filled the shells with diagnostic gas before coating with PVA. We now permeate the capsules with the diagnostic gas after the GDP coating.

A number of changes reduced debris. We devised a new PVA fill/injection device that has reduced debris and decreased the number of shells that plug the injection capillary. In addition, we now inspect our polystyrene mandrels more intensely for cleanliness. Also, we have begun to size the polystyrene shells by dry sieving (rather than washing the shells through the sieves with isopropanol) which may result in cleaner mandrels. The procedure for cleaning the capillaries and stop wires has been expanded and now includes a sonic cleaning step.

Our process improvement efforts increased product quality while our process yield is double that of the previous year.

Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
ICF TARGET CAPSULE DESIGN

A layer of polyvinyl alcohol (PVA) entraps the fill gases in the capsule.

-3 \mu m PVA

-50 \mu m CH

-3 \mu m polystyrene

0.1 atm Argon & DD or DT

GENERIC ICF CAPSULE

ICF TARGET PRODUCTION PHASES IN FY93

Polystyrene Shell Drop Tower → Argon Permeation → PVA Coating Drying Tower → Glow Discharge Polymerization (GDP) Coater
THE SHELLS WERE PERMEATED WITH DIAGNOSTIC GAS BEFORE PVA COATING

BARE POLYSTYRENE SHELLS WERE FILLED WITH ARGON

1. Chamber is evacuated
2. Chamber is filled to desired argon pressure
3. Argon permeates into uncoated PS shells

Shells in capillary

PVA solution

THEN AN AQUEOUS PVA SOLUTION ENTRAPPED THE ARGON IN THE SHELLS

PVA solution

THE SHELLS WERE INJECTED INTO THE DRYING TOWER WITH A SYRINGE

The capillary filled with shells immersed in PVA were attached to a PVA filled syringe and the shells were injected into the drop tower.

Capillary tube filled with shells

Syringe filled with PVA solution

Drop tower heated to ~110 °C

Polystyrene shell in droplet of PVA solution

Polystyrene shell with solidified PVA coating

Shell collection plate
HISTORICALLY THE PVA COATING PROCESS HAS BEEN OF LOW YIELD

DEFECTS IN PVA COATED SHELLS
FIRST YEAR OF OPERATION (FY93)

Strings and debris were the two major defects

Frequency

Strings Small Bubbles Mangled Large Bubbles PVA Fill Tool Mark Dents Gross Debris Glued on

WE DECREASED STRING FORMATION BY MODIFICATION TO OUR GAS FILL PROCEDURES

Formerly we permeated the polystyrene shell with diagnostic gas (Ar) before coating with PVA.

During this procedure the bare polystyrene shells were kept under vacuum for an extended period which caused the shells to stick together forming “strings”.

Argon fill pressures were limited to < 1 atm.

We now permeate the shells with the diagnostic gas after GDP coating.

Since the bare polystyrene shells are no longer evacuated, our new procedure has resulted in a marked decrease in strings from the PVA coating process.

We are now capable of fill pressures > 1 atm.

The new method results in more reliable gas fill pressures. We have reduced the pressure variation from shell-to-shell to << 10%.
GDP-coated shells are placed in an eggcrate in a permeation apparatus. The chamber is pumped down and backfilled with the specified diagnostic gas at the required pressure.

The permeation apparatus is then heated in an oven for 24 hours at 142°C.

ICF TARGET PRODUCTION PHASES IN FY95

- Polystyrene Shell Drop Tower
- PVA Coating Drying Tower
- Glow Discharge Polymerization (GDP) Coater
- Argon Permeation
REDUCTION IN DEBRIS HAS BEEN ACHIEVED BY THE FOLLOWING PROCESS MODIFICATIONS

1. A New PVA Fill / Injection Device has been introduced which minimizes contamination of the PVA solution.

2. The ends of the capillaries are plugged while grinding the tips resulting in cleaner capillaries.

3. The procedures for cleaning the capillaries and stop wires have been expanded to include a sonic cleaning step.

4. The polystyrene mandrels are inspected for debris by the characterization group before PVA coating.

5. When sizing the polystyrene mandrels, the shells are dry-sieved as opposed to washing the shells through the sieves with isopropanol.

MODIFICATION TO PVA FILL / INJECTION METHOD

Previously a syringe was used to fill the capillaries with PVA solution and inject the shells into the drying tower.

During a run of several capillaries the syringe had to be refilled repeatedly subjecting the reservoir of PVA solution to contamination.

We have introduced a new PVA fill injection device which remains sealed after the solution is filtered directly into it.

Contamination of the PVA solution with debris is minimized since there is no further direct contact with the solution reservoir during a run series.
NOW THE SHELLS ARE INJECTED INTO THE DRYING TOWER WITH A NEW PVA FILL/INJECTOR DEVICE

Squeezing the bulb forces the warm PVA solution into the shell-filled capillary.

Squeezing the bulb forces the shells and PVA solution into the drop tower.

NEW CAPILLARY PREPARATION AND CLEANING PROCEDURES

1. A very small amount of petroleum jelly is placed in the tips of the capillaries before grinding the tips to a taper.

2. The capillaries are rinsed forcefully with methanol and then burned in a muffle oven at 400°C for two hours.

3. The capillaries and stop wires are sonicated in double filtered water for 90 minutes.

4. The capillaries and stop wires are rinsed with double filtered water and blown dry with filtered nitrogen.

5. The capillaries and stop wires are further dried in a 40°C drying oven for 2 hours.
FUTURE PROCESS IMPROVEMENTS WILL FOCUS ON BUBBLES, SMALL DEBRIS AND DEWET

DISTRIBUTION OF PVA COATING DEFECTS Jul-Sep 94

BY REDUCING STRINGS AND SMALL DEBRIS WE HAVE INCREASED OUR PVA COATING PROCESS YIELD

We have increased our yield while our acceptance criteria tightened and as we produce higher quality targets.
We have developed an apparatus that stably levitates a single microshell using an acoustic standing wave. The apparatus consists of an ultrasonic generator (homogenizer/mixer) and a reflector that is adjusted to produce a standing acoustic wave. At sufficient acoustic power, microshells stably levitate at a position slightly above the planes of minimum pressure where the acoustic force acting on the shell exactly balance gravitational force. We have investigated coating a shell with aqueous PVA solutions with this apparatus. Difficulties were encountered in metering the minute quantities of liquid (in the range of 40 nl) needed for the coating. It was found that an aerosol mist injected in the acoustic field would envelop the levitated microshell, provided it was wetable. Drying was accelerated using infrared heating. Surface finish of the dry coating was occasionally good, although thickness uniformity was distorted by the acoustic field. A method for random tumbling of the shell is needed to achieve uniform coatings.

*Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Development of Acoustic Levitation for PVA Coating

Robert A. Reibold, Stephan A. Letts and Robert C. Cook

Lawrence Livermore National Laboratory
Livermore, CA 94551
Levitation techniques offer many benefits for the production of ICF targets

- **Multiple coatings capability.**
  - Single shell processing
  - Diagnostic materials
  - PVA gas barrier
  - CH ablator

- Permits the **step-by-step** characterization of the target during its lifetime.

---

**Acoustic levitation: Historical development of acoustic levitation techniques**

- Electromagnetic devices - removal of suspended matter through flocculation of aerosols - H.W. St. Clair, 1941
- Piezoelectric (PZT) materials - materials improvements lead to piezoelectric acoustic drop holders for single droplet atomization studies - A.R. Hanson, 1964
- U.S. Space Program - containerless processing in zero gravity environments - T.G. Wang, R.R. Whymark, 1974
- Polymer coatings for ICF targets - stalk mounted glass spheres by Wang in 1982, and freely levitated PS mandrels by Mark C. Lee in 1984
- Droptower experiments precluded further attempts to improve acoustic techniques designed for coating applications - Eben Lilley, 1986
Poly(vinyl alcohol) is applied to PS mandrels using droptowers

1. Vacuum PS mandrels into a glass capillary tube. (Shells - 0.5 mm O.D., tube 0.36 mm I. D.)

2. Fill tube with 10% polyvinyl alcohol aqueous solution.


4. Shells dry in 5 meter column at 100°C. Final PVA coating is 3 to 4 um thick.

Droptower technology has limitations

- Polystyrene shell formation
  - < 1% recovery of target quality shells

- PVA coating
  - 3 to 5% recovery of target quality coated shells

- Trouble producing larger shells

- Identity of the individual capsule is lost

- Not capable of providing the projected 100's of PS mandrels needed to meet future demands
Acoustic levitation offers some possibilities for ICF target production

- Stable positioning of PS mandrels in an acoustic field
- Application of coating materials
- Rotate and/or oscillate a levitated specimen
- Heat the levitated specimen uniformly

Acoustic Levitation Project

OBJECTIVE: Develop the capability of taking a single, well characterized plastic mandrel, and applying a smooth symmetric poly(vinyl alcohol) coating to it while the capsule is levitated in an acoustic field.
A commercial sonicator was converted to an acoustic levitator for coating purposes.

Acoustic levitation requires a high intensity sound source and stationary compression waves.

Sound Pressure Distribution

Acoustic energy wells showing sound pressure distribution of a standing wave field and the near field.
By manipulating the reflector centering of the PS mandrel can be achieved

- Centering forces can be used to control positioning of the PS mandrel

No centering attempted  Centering forces applied

SEM photos reveal "pits" in a thin PVA coating when applied as a dilute solution

- Thin PVA coating was dried at room temperature

PVA coated PS mandrel at 150X magnification

PVA surface showing "pits" in coating at 1000X magnification

Cross section of PS mandrel with PVA coating at 1000X magnification
PVA can be applied by "misting" solution from the transducers vibrating surface.

- Coating uniformity is affected by gravity.

![Thin PVA coating < 0.5 μm](image1)

![Heavy PVA coating on bottom](image2)

Concentrated PVA solution when dried show no effects of "pitting" on the PVA surface.

- PVA was applied to stalk mounted PS mandrels while rotating in the acoustic field - dried with infra-red heat.

![PVA coated PS mandrel on a glass stalk at 80X magnification](image3)

![SEM mounted PVA coated PS mandrel at 150X magnification](image4)

![Smooth PVA surface at 500X magnification](image5)

![Cross section showing 5 μm PVA layer at 5000X magnification](image6)
Summary:

- Created a contact free environment
- Shown that levitation of 500 μm PS shells is possible
- Applied PVA coatings
- Dried coatings using infra-red heat
- Produced a smooth surface

Some things we have learned:

- From the literature:
  - Major levitation problems have been solved - need to be applied at the sub millimeter scale.
  - Opposed sets of transducers eliminate effects due to temperature at the center pressure node.
  - Specimens can be rotated and/or oscillated by modifying the phase angles and/or the amplitude of the acoustic field.

- From our Experience:
  - The current levitation system is inadequate for production PVA coatings.
  - A delivery system for dispensing microliter and nanoliter quantities of PVA solution is needed.
  - An efficient PVA drying system is needed designed to heat water only having minimal effect on PVA and PS.
Polyvinyl alcohol (PVA) is used as a gas-barrier to hold deuterium and diagnostic gases (such as argon) inside the hollow, spherical shells used as laser fusion targets. A PVA layer several microns thick will, at room temperature, hold on the order of 50 atmospheres of deuterium in the shells for several hours, a time sufficient for laser experiments. However, this thickness of PVA permanently entraps the argon. Normally, the argon is entrapped when the PVA coating is applied. We have developed a procedure whereby argon is permeated through the PVA layer of completed shells to fill them to specified fill pressures (typically 0.1 to 1.0 atm). The shells are placed in a sealed fill tube pressurized with the desired amount of argon. The tube is then heated to approximately 140°C and kept at temperature for 24 hours until the argon pressure in the shell reaches equilibrium. An X-Ray Fluorescence (XRF) system is used to measure the argon content of the shells so that permeation coefficients can be calculated. We have found that half-millimeter shells coated with 3 μm of PVA can be filled with argon in one day. This procedure has been used to produce: argon fills in excess of 1 atmosphere, uniform shell-to-shell argon content, removal of residual gases (i.e., air and water vapor) from the shells, and calibration standards for XRF measurement of shell argon content.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
ICF capsules are filled with argon for temperature diagnostic purposes.

A layer of polyvinyl alcohol (PVA) entraps argon in the capsule.

Generic ICF capsule:
- ~3 µm PVA
- ~3 µm polystyrene
- ~50 µm CH
- 0.1 atm Argon
Previously, bare polystyrene shells were filled with argon.

1. Chamber is evacuated.
2. Chamber is filled to desired argon pressure.
3. Argon permeates into uncoated PS shells.

An aqueous PVA solution entrapped the argon in the bare shells.
THE LIQUID-COVERED SHELLS WERE INJECTED INTO A DROP TOWER TO SOLIDIFY THE PVA

- Capillary Tube Filled With Shells
- Syringe Filled With PVA Solution
- Drop Tower Heated to ~120 °C
- Polystyrene Shell in Droplet of PVA Solution
- Polystyrene Shell With Solidified PVA Coating
- Shell Collection Plate

THE PREVIOUS ARGON FILL TECHNIQUE HAD SOME SHORTCOMINGS

- We found up to a 20% pressure variation from shell-to-shell.
- The argon fill pressure was limited to < 1 atm.
- Gases (air, water vapor) in the PVA solution permeated into the shells.
- Cleanliness of PVA solution was difficult to maintain.
IN OUR TECHNIQUE, WE PVA-COAT SHELLS BEFORE ARGON IS INTRODUCED

Squeezing the bulb forces the PVA solution into the shell-filled capillary

THE SHELLS ARE THEN INJECTED DIRECTLY INTO THE DROP TOWER
THE PVA-COATED SHELLS ARE PLACED IN AN EGGCRATE IN A PERMEATION APPARATUS

THE PERMEATION APPARATUS IS PRESSURIZED THEN HEATED IN AN OVEN FOR 24 HOURS
WE HAVE MEASURED ARGON'S PERMEATION HALF-LIFE AT A VARIETY OF TEMPERATURES

Permeation Half-life vs. Temperature

(440 μm I.D. PS Shell Coated With ~3 μm of PVA and ~50 μm of Polymer)

ARGON PERMEATION THROUGH PVA AT ELEVATED TEMPERATURES IS NOW ROUTINE

- We have filled and delivered composite capsules with 0.05, 0.1, and 10 atm of diagnostic gases.
- We have filled composite capsules with a 100:1 mixture of neon and argon.
- We have reduced the pressure variation from shell-to-shell to << 10%.
- We now can use witness shells to verify the argon content in shells that XRF systems cannot analyze (i.e., germanium-doped layers).
DEUTERIUM AND TRITIUM PERMEABILITY THROUGH ALUMINUM FILMS AT HIGH PRESSURES

W. T. Shmayda
Ontario Hydro Technologies
800 Kipling Ave., Toronto, ON, Canada, M8Z5S4

H. Kim, M. Wittman, and R. Gram
University of Rochester Laboratory for Laser Energetics
Rochester, NY, USA, 14623

The performance of polystyrene microshells filled with equimolar mixtures of deuterium-tritium will be assessed at the University of Rochester OMEGA facility in 1995 as part of their Inertial Confinement Fusion program using laser drivers. These targets consist of hollow, thin-walled, plastic spheres overcoated with an aluminum layer and charged with DT gas to several tens of atmospheres. The aluminum overcoat acts as a permeation barrier to restrict DT loss from the shell and as a radiation barrier to optimize the laser energy coupling with the target. The stability of the aluminum coating in the presence of tritium is questionable since beta energy arising from tritium decay can induce radiation damage within the bulk and at the surfaces of the 40 to 120 Å thick coatings. A degradation in the aluminum film can lead to a dramatic increase in tritium egress from the targets with a concomitant impact on the burn physics. To address these concerns an experimental program has been initiated to measure the dependence of hydrogen isotope permeability through the aluminum coatings on thickness, on aging in air and on radiation damage.

This paper will describe the deuterium and tritium facilities used to charge the microspheres and subsequently to monitor the permeant loss from the spheres. Deuterium permeation curves will be presented for un-tritiated aluminized shells. Diffusivity values and their dependence on film thickness, aging in air and hydrogen, surface morphology, crystal orientation and crystal structure will be provided and discussed. Preliminary measurements on the impact of radiation damage on permeation will be presented.

This work was supported by the U.S. Department of Energy Office of Inertial Confinement Fusion under Cooperative Agreement No. DE-AP03-92SF19460 and by the Canadian Fusion Fuels Technology Project under contract number CI-776-92399-3109-994.
Capsules must contain their fuel with a half life greater than 20 hours to be useful. This is attained by adding a gas barrier layer to composite shells. Leaks in this layer are not detectable except by direct measurement of hydrogen leak rate. We have developed a procedure which can detect excessive leak rates and cull defective shells rapidly and without excessive shell handling. Shells are filled with H₂ to 50 atmospheres. The fill-crate has an slit in it so that they can be interferometrically examined in situ. The shells' leak rate can be determined by two measurements of fringe position over a period of 4 hours. The fringes should move 1/2 to 1 fringe spacing in that time. A larger shift indicates an unacceptable leak. A fixed fringe indicates a leak so large that the capsule was empty when first examined. The design and effectiveness of this procedure and the modified fill-crate will be presented.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
USE OF FILL-CRATE IMPROVES RELIABILITY AND EFFICIENCY OF HYDROGEN OUT-GASSING RATE MEASUREMENTS

Martin L. Hoppe
General Atomics
IMPROVED INTERFEROMETRIC METHOD FOR D-D HALF-LIFE DETERMINATION HAS SEVERAL KEY ADVANTAGES

**Improved Method**

- Shells are Readily Characterized While Still in Filling Crate
- Decreased Probability of Loss of, or Damage to, Capsule
- Increased Clarity of Fringes, Thus Increased Precision in Data
- Increased Efficiency - Fewer Man-hours Required

ORIGINAL INTERFEROMETRIC METHOD FOR D-D HALF-LIFE DETERMINATION HAS SEVERAL DRAWBACKS

**Original Method**

- Repeated Handling of Each Shell was Required
- Increased Probability of Loss of, or Damage to, Capsule
- Fringes Distorted by XRF Film, Thus Decreased Data Precision
- Time Consuming
DETERMINATION OF D-D HALF-LIFE BY FRINGE SHIFT

Standard Size Capsule at Room Temperature

Half-Life Vs Fringe Shift @4hr. With 50 Atm Fill

Fringe Shift is Change in Fringe Count at Time ~0 Relative to Fringe Count After 4 hrs. at Room Temperature

A Third Fringe Count is Required for Confirmation

BASIC INTERFEROMETRIC PROCEDURE FOR D-D HALF-LIFE DETERMINATION

- Capsules are Filled to 50 Atm with D-D
- Optical Path Length Thru Shell Measured by Interferometry
- Changes in Optical Path Length are Monitored as a Function of Time to Determine Out-gassing Rate (1/2-Life) of D-D
- For a Standard Size Composite Capsule, 1 Fringe Shift Equals ~10 Atm Loss in D-D Pressure
IN IMPROVED METHOD SHELL FRINGES ARE READILY MEASURED WHILE STILL MOUNTED IN FILL-CRATE

ORIGINAL METHOD REQUIRED SHELLS TO BE MOUNTED IN XRF SAMPLE HOLDERS AFTER D-D FILLING
ENLARGED VIEW OF PROTOTYPE FILL - CRATE CELLS

AI (7075 GRADE) FILL - CRATE DIMENSIONS (BODY)

Overall Dimensions: 51.0 mm x 3.6 mm x 2.2 mm

Top Views

- 0.35 mm slit
- 0.40 mm hole
- 0.80 mm well 1.20 mm deep

48 mm center-to-center

End view

Threaded screw hole for 1/8" #0-80 screw

0.35 mm wide slit

0.40 mm diameter through holes centered in wells

Threaded screw hole for 1/8" #0-80 screw

25 evenly spaced, tapered "wells" at 1.20 mm deep by 0.80 mm diameter
FILL- CRATE LID DIMENSIONS

51.0 mm x 3.6 mm x 1.0 mm
48 mm center to center
45 mm
0.35 mm wide slit
Unthreaded hole to allow for #0-80 screws to pass thru

ACKNOWLEDGEMENTS

David A. Steinman

U.S. Dept. Of Energy under Contract No. DE-AC03-91SF18601
Recently requested task work calls for CH coatings doped with chlorine. CH polymer produced by plasma polymerization has been found to take up oxygen.\textsuperscript{1,2} Storage of newly-formed polymer in chlorine may result in chlorinated rather than oxygenated polymer. Experiments are now in progress in which CH-coated capsules stored in chlorine gas are tested for total chlorine uptake by x-ray fluorescence and for chlorine concentration homogeneity by EDXS. The results of these experiments and the prospects for using this technique to produce chlorine-doped CH coatings is reported.

\textsuperscript{*}Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
\textsuperscript{1}R. Turner, Lawrence Livermore National Laboratory Memo No. TST 94-064 (1994).
**PROCEDURE**

Undoped polystyrene (PS) shells were coated with 3 or 60 μm of CH by plasma polymerization.

The CH-coated shells, plus some uncoated PS shells, were loaded into glass capillary tubes, which were then placed in a six-position manifold.

Air exposure was minimized (~1 hr.).

The manifold was evacuated and backfilled with chlorine gas (99.9% minimum purity).

Shells were stored in the dark in 1.1 atmospheres of chlorine gas at room temperature.

At intervals, shells were removed, inspected, measured and tested for chlorine content by X-ray fluorescence (XRF).

Shells with thick CH coatings were broken and tested for chlorine concentration homogeneity by electron probe X-ray microanalysis (EPMA) (incident beam energy = 10 keV) and energy-dispersive X-ray spectrometry (EDXS).

---

**INITIAL RESULTS WERE ENCOURAGING**

Results for 3 μm CH on 3 μm PS:

<table>
<thead>
<tr>
<th>Days of Cl Storage</th>
<th>Number of Shells</th>
<th>Chlorine Counts/ sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (control)</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>3</td>
<td>1149</td>
</tr>
<tr>
<td>2</td>
<td>3</td>
<td>1160</td>
</tr>
<tr>
<td>4</td>
<td>3</td>
<td>1245</td>
</tr>
</tbody>
</table>

These results imply 7–8 atom % chlorine uptake by the CH, assuming that all chlorine resided within the CH layer.

**This was not the case.**
WE FOUND CHLORINE IN THE POLYSTYRENE

X-ray fluorescence readings for uncoated polystyrene shells stored in chlorine gas approached those for the 3 μm CH.

<table>
<thead>
<tr>
<th>Days of CI Storage</th>
<th>Number of Shells</th>
<th>Chlorine Counts/sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 μm CH on 3 μm Polystyrene</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>3</td>
<td>1245</td>
</tr>
<tr>
<td>42</td>
<td>2</td>
<td>1466</td>
</tr>
</tbody>
</table>

3 μm Polystyrene Alone

<table>
<thead>
<tr>
<th>Days of CI Storage</th>
<th>Number of Shells</th>
<th>Chlorine Counts/sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>35</td>
<td>5</td>
<td>1299</td>
</tr>
</tbody>
</table>

The results for uncoated polystyrene indicate that the polystyrene alone took up 7.5 atom % chlorine.

If this was also true of the CH-coated polystyrene, almost none of the chlorine is in the CH layer.

THICK CH RESULTS INDICATE THAT CH DOES TAKE UP CHLORINE

Thick CH on 3 μm PS:

<table>
<thead>
<tr>
<th>Days of CI Storage</th>
<th>Number of Shells</th>
<th>Chlorine Counts/sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>60 μm CH on Polystyrene</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0 (control)</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>21</td>
<td>2</td>
<td>4386</td>
</tr>
<tr>
<td>35</td>
<td>1</td>
<td>3094</td>
</tr>
</tbody>
</table>

50 μm CH on Polystyrene

<table>
<thead>
<tr>
<th>Days of CI Storage</th>
<th>Number of Shells</th>
<th>Chlorine Counts/sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>35</td>
<td>1</td>
<td>3007</td>
</tr>
</tbody>
</table>

In each case the number of chlorine counts is significantly higher than that for uncoated PS or thin CH.
EPMA was done at intervals along the broken shell wall.

Chlorine was present throughout the shell wall.

Positions 0 and 13 are the inner and outer surfaces, respectively.
DROPLETS APPEARED WITHIN THE THICK SHELLS

CHLORINE EXPOSURE INCREASED SHELL DIAMETERS

Thin CH (3 μm) ID's increased by 7-8%.

<table>
<thead>
<tr>
<th>Presoak ID (μm)</th>
<th>Day</th>
<th>Postsoak ID (μm)</th>
<th>Number of Shells</th>
<th>Δ ID (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>442</td>
<td>1</td>
<td>471</td>
<td>3</td>
<td>6.6</td>
</tr>
<tr>
<td>(average)</td>
<td>2</td>
<td>470</td>
<td>3</td>
<td>6.3</td>
</tr>
<tr>
<td>4</td>
<td>42</td>
<td>473</td>
<td>3</td>
<td>7.0</td>
</tr>
<tr>
<td>42</td>
<td>46</td>
<td>476</td>
<td>2</td>
<td>7.7</td>
</tr>
</tbody>
</table>

Thick CH (60 μm) OD's increased by 7-8%.

<table>
<thead>
<tr>
<th>Presoak OD (μm)</th>
<th>Day</th>
<th>Postsoak OD (μm)</th>
<th>Number of Shells</th>
<th>Δ OD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>564</td>
<td>21</td>
<td>604</td>
<td>2</td>
<td>7.1</td>
</tr>
<tr>
<td>565</td>
<td>35</td>
<td>611</td>
<td>1</td>
<td>8.1</td>
</tr>
</tbody>
</table>

Uncoated PS OD's increased by 12%.

<table>
<thead>
<tr>
<th>Presoak OD (μm)</th>
<th>Day</th>
<th>Postsoak OD (μm)</th>
<th>Number of Shells</th>
<th>Δ OD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>446</td>
<td>35</td>
<td>501</td>
<td>6</td>
<td>12.4</td>
</tr>
</tbody>
</table>
CONCLUSIONS

Plasma polymerized CH coatings take up chlorine when stored in chlorine gas.

Uncoated polystyrene mandrels take up 7-8 atomic % chlorine.

The diameters of the polystyrene mandrels, as well as those of CH-coated shells, increase with chlorine exposure. It may be possible to compensate for dimensional changes.

While chlorine soaking can be used to produce chlorine-doped CH, the chlorine acquired by the inner mandrel limits the usefulness of this technique.

Furthermore, tailoring the chlorine content of the CH layer to customer specifications may be difficult.

Many important issues remain to be addressed, including questions about absolute chlorine content, stability, physical and mechanical properties, uniformity and possible inner surface contamination.

We recommend that further work on this technique not be done unless alternative means of producing chlorine-doped CH coatings work less well than this method.

QUESTIONS FOR FURTHER STUDY

Many questions about the chlorine uptake of CH shells stored in chlorine gas remain. Among these are:

How much chlorine is present in the CH coating alone?

How reproducible is the chlorine distribution within the shell wall?

What is the minimum time required to obtain an acceptably uniform chlorine distribution?

What are the droplets within the thick shells after the chlorine soak?

Can this substance be removed without damaging the capsule?

How predictable are the dimensional changes that occur with chlorine soaking?

Do the dimensional changes adversely affect capsule uniformity?

How does the incorporation of chlorine affect the physical and mechanical properties of the capsule?

Does the chlorine concentration remain stable over time?

How is the chlorine incorporated into the PS and into the CH?
PROCESS ISSUES ASSOCIATED WITH MAKING FOAM MICROSHHELLS AT LLNL

G.E. Overturf, S.R. Buckley and R.C. Cook, LLNL
M.R. McClellan, WJSA

University of California
Lawrence Livermore National Laboratory
P.O. Box 808, Livermore, CA 94551

This poster is a venue for informal discussion of current process developments related to the cryogenic foam shell target developments. Topics presented may include microshell droplet generation, foam shell characterization, solvent exchange techniques and gelation issues.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Process issues with Foam Shells

George Overturf  
Robert Cook  
Steve Letts  
Steve Buckley  
Mike McClellan, WJSA  
Diana Schroen-Carey, WJSA

Tenth Target Specialist's Meeting  
Taos, New Mexico  
February 7, 1995
Resorcinol/Formaldehyde Reactions

In a basic carbonate solution, resorcinol forms the resocinolate anion which is more strongly ortho para directing than the neutral diphenol because a full negative charge is present.

Nucleophilic attack by the phenolate anion on formaldehyde yields a hydroxymethyl derivative of resorcinol.

Loss of an OH group causes the hydroxymethyl derivative to form a quinone-like intermediate which subsequently reacts with a phenoxide or another hydroxymethyl resorcinol to form methylene and methylene ether bridges.

The acid catalyzed reaction proceeds by a S,2 displacement of water from a protonated hydroxymethyl group by the nucleophilic oxygen on an unprotonated hydroxymethyl group.

Polymernization of resorcinol with formaldehyde followed by acid catalysis produces a crosslinked structure.

These crosslinked polymers nucleate into R/F clusters which are 70-100Å in diameter. The clusters aggregate together to form chains which give the "string of beads" morphology.
The resorcinol / formaldehyde solution is weighed then heated at 70°C to promote gelation.

Resorcinol
Na₂CO₃
Water
Formaldehyde

Combine

Heat
Stir

Benzoic acid

Benzoic acid is added after 1h to increase the rate of gelation.

Foam Shells are produced using a droplet generator.

Shell diameter and wall thickness are controlled by fluid flow rates.

Solvent (water), monomer, initiator

Low-density foam shell

polymerize
exchange solvents and supercritically dry using CO₂
A triple orifice droplet generator is used to produce resorcinol / formaldehyde shells having a diameter of 2 - 3mm and 100 - 200μm walls.

<table>
<thead>
<tr>
<th>10ml syringe</th>
<th>Inner phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oil phase to inner orifice @ 25μl / min</td>
<td>18g dibutylphthalate</td>
</tr>
<tr>
<td>RF solution to middle orifice @ 200μl / min</td>
<td>2g toluene</td>
</tr>
<tr>
<td>50ml syringe</td>
<td>20g stripping fluid</td>
</tr>
</tbody>
</table>

Polymer solution:
- 2.36g resorcinol
- 60ml H₂O
- 5ml Na₂CO₃
- 3.34ml HCOH

Stripping phase:
- 900g dibutylphthalate
- 100g toluene
- 0.50g PEG 400

Rotary evaporator set to 30 rpm

Water bath @ 60°C
Characterization

Interferometry*

Optical path length distorted by polymer in wall, giving typical uniformity and polymer thickness information.

\[
\frac{\Delta \text{ Optical Path}}{(n_{\text{polymer}} \cdot \eta_{\text{air}})} = \text{Polymer Thickness}
\]

\[
\frac{(\text{Polymer Thickness})(\text{Intrinsic Density})}{\text{Foam Wall Thickness}} = \text{Foam Density}
\]

*Technique from R. Stephens, General Atomics

After being formed, resorcinol gelled shells are processed through a series of solvent exchanges

Isopropyl Alcohol

Acetic acid

Isopropyl Alcohol

CO₂ supercritical dryer
Transparent RF shells have been produced in the size range of interest for NIF.

Shell diameters to 2 mm.  Wall thickness to 400\(\mu\)m.  Foam densities approaching 50 mg/cc.

Foams have been cast as discs and spin coated onto glass tubing for cryogenic studies.

R/F and trimethacrylate are molded against quartz windows in copper discs which have 100\(\mu\)m recesses.

RF solution inside of glass tubing.

High speed motor.

RF spin coated onto glass tubing.
Photos of an R/F spin coated quartz tube

6X
R/F inside of Glass tube

26X
Closeup of crosssectioned wall
DEVELOPMENT OF LOW DENSITY, LOW ATOMIC NUMBER FOAM SHELL FOR LASER FUSION TARGET

M. Takagi, T. Norimatsu, Y. Izawa, and S. Nakai
Institute of Laser Engineering, Osaka University
2-6, Yamada-Oka, Suita, Osaka 565 Japan

Recent progress in laser fusion research requires cryogenic foam targets to produce a thick uniform liquid fuel layer. The requirements for the foam shell are as follows: The diameters and the thickness of the foam shells are 1 to 3 mm and 10 to 100 μm, respectively. The density and the cell size of the foam layer must be <50 mg/cm³ and <0.5 μm, respectively. The wall thickness and the surface finish of the gas barrier are 10 to 30 μm and 20 mm, respectively. The foam must be optically transparent to allow optical diagnostics of the fuel layer through a 200 μm thick foam layer in total.

We have been developing the emulsion method to fabricate large, uniform and low density foam density foam shell. The process is based on polymerization of monomers in a density matched water/oil/water (W₁/O/W₂) emulsion. W₁ and W₂ are water phases, which include small amount of emulsion stabilizer or surfactant in some cases. O is a monomer solution with polymerization initiator. A mixture of diethyl phthalate and dibutyl phthalate is used as the O phase solution for density-matching with W₁ phase solution. Such emulsion is produced by a droplet generator in water (W₂). Then the emulsion is heated up to start the gelation in the O phase. After the reaction is completed, the O phase solution is replaced with ethanol and dried in a super critical drying apparatus.

In addition to trimethylolpropane trimethacrylate (TMPT) foam shells¹ made by thermal polymerization using AIBN, we have recently developed ethyleneglycol dimethacrylate (EGDM) foam shell made by redox polymerization. In this case the redox catalysts were added in O and W₂ phases solution. The polymerization starts and proceeds at room temperature after mixing them together.

Low density foam shells with high optical transmittance were successfully fabricated using highly concentrated polymerization initiator (AIBN) for TMPT and using EGDM polymerized with redox catalysts at room temperature. With the droplet generator, 0.1-2.8 mm diameter foam shells with 10-160 μm thick walls were produced in small dimensional variation. In TMPT-foams polymerized by AIBN, the transmittance of visible light was greatly improved with increase in the concentration of AIBN. The foam density was 50-70 mg/cc. In the redox-polymerization of EGDM, the cell size of the foam was mostly less than 0.6 μm and the foam density was 60-70 mg/cc. Measurement of the EGDM foam thickness above 160 μm was possible by a normal optical microscope. This fact allows optical characterization of fuel layer in cryogenic targets.

Low density micro cellular foams containing dispersed high atomic number material have been produced. The work done by R. Simandl et al., using phase inversion of a binary solvent system and 4-methyl 1-pentene to produce microporous polymer foams was successfully duplicated. To enhance the diagnostic properties of the material, molybdenum, a high Z material, was dispersed throughout the foam. We have been able to incorporate the molybdenum into the foam structure while maintaining a relatively low bulk density. Foam samples with initial molybdenum concentrations up to 25 wt % and have bulk densities as low as 15 mg/cc have been produced. Still higher molybdenum contents and lower foam density are being pursued. The uniformity of the molybdenum dispersion has not yet been quantitatively characterized but does vary from sample to sample. Further work is currently being done to improve the uniformity of the molybdenum dispersion throughout the foam and to further lower the densities of the resulting foams.

*Work supported by GA Fusion IR&D.
OBJECTIVE

To produce low density polymer foams with a high Z dopant to improve diagnostic capabilities of currently used polymer foam liners.

GOALS

• To produce a low density polymer foam doped with molybdenum.

• To incorporate this dopant inherently and uniformly throughout the structure of the foam sample.

• To determine the amount of dopant that can be incorporated into the foam structure while maintaining low density and some structural integrity.

• To determine the processing conditions to make low density doped foams with uniform dopant dispersion and small cell size.
The Chemical System*

- **Polymer:** TPX®
  - poly(4 methyl-1-pentene)
  - linear CH polymer
  - density: 0.83 gm/cm³

- **Solvents:**
  - Durene (re crystallized from ETOH)
  - Naphthalene
    - Durene / Naphthalene Ratio 60/40 (wt%)

- **Process:** Phase Inversion from the Binary Solvent/Non solvent System
  - Mix polymer and solvents together in a glass tube.
  - Heat contents of tube to at least 180°C to dissolve TPX® polymer.
  - Cool tube slowly through gelation.
  - Use unidirectional cooling to cool the sample to ice temperature.
  - Use sublimation to remove the solid solvents. CO2 soxhlet extraction provided no advantage.


* P. Apen, Los Alamos National Laboratory, Discussion, April 1994.
Measurement Methods

- **Density** = mass/volume
  - MASS is measured with a Sartorius micro balance.
  - Volume is calculated from measurements of the sample dimensions obtained using calipers. **This limits samples to those with a simple geometry.**

- **Foam Morphology**
  - Scanning electron microscopy is used to study the structure of the foams.
  - Visual Observation allows the detection of highly anisotropic structures.

- **Molybdenum Uniformity**
  - Visual observation of the sample color after cooling is used as an initial determination of whether the molybdenum is uniformly dispersed throughout the sample.
  - Back scatter electrons, from the scanning electron microscope, can be used qualitatively to create an x-ray map of the foam surface. Due to the rough surface of the foam, the electron map is not sensitive enough to be used quantitatively.

- **Cell Size**
  - Calipers were used to measure the cell dimensions of the doped foam samples using micrographs from the scanning electron microscope.
  - Average cell sizes and standard deviations, using at least twelve measurements for each sample, were calculated.
Figure 1. Gradient Cooling System

Density

<table>
<thead>
<tr>
<th>Label</th>
<th>Theo $\delta$</th>
<th>%TPX</th>
<th>%Mo ave $\delta$(mg/cc)</th>
<th>Wt% Mo</th>
<th>%Mo/%TPX</th>
<th>Average Cell Size (um)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>12/9/94</td>
<td>6.90</td>
<td>0.65</td>
<td>0.13</td>
<td>10.50</td>
<td>11.69</td>
<td>0.20</td>
<td>5 ± 2 light grey</td>
</tr>
<tr>
<td>12/14/94</td>
<td>16.60</td>
<td>0.63</td>
<td>1.23</td>
<td>13.90</td>
<td>25.26</td>
<td>1.96</td>
<td>65 ± 31 grey/green very anisotropic</td>
</tr>
<tr>
<td>12/16/94</td>
<td>15.80</td>
<td>0.63</td>
<td>1.14</td>
<td>14.45</td>
<td>25.01</td>
<td>1.82</td>
<td>37 ± 15 grey</td>
</tr>
<tr>
<td>12/19/94A</td>
<td>12.10</td>
<td>0.72</td>
<td>0.66</td>
<td>17.50</td>
<td>22.00</td>
<td>0.91</td>
<td>25 ± 6 grey/green</td>
</tr>
<tr>
<td>12/20/94B</td>
<td>12.60</td>
<td>0.69</td>
<td>0.73</td>
<td>13.65</td>
<td>22.89</td>
<td>1.09</td>
<td>19 ± 7 grey/blue</td>
</tr>
<tr>
<td>1/4/95</td>
<td>3.80</td>
<td>0.44</td>
<td>0.00</td>
<td>6.85</td>
<td>0</td>
<td>NA</td>
<td>15 ± 7 white, no quench, stuck to tube,</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>pieces broke when removing</td>
</tr>
<tr>
<td>1/6/95</td>
<td>5.60</td>
<td>0.65</td>
<td>0.00</td>
<td>7.70</td>
<td>0</td>
<td>NA</td>
<td>11 ± 5 white</td>
</tr>
</tbody>
</table>

Figure 2. Tabulation of Density Results
Figure 3a. Well connected polymer webbing: Cooling rate in gelation region @ 0.5°C/min. Sample density @ 4.4 mg/cc, Wt% Mo @ 25%, %TPX,%Mc @ 0.54.

Figure 3b. "Rose like" structure: Cooling rate in gelation region @ 2.3°C/min. Sample density @ 15.9 mg/cc, Wt% Mo @ 25%, %TPX,%Mc @ 0.5.
Figure 4a. Scanning electron microscope photo of a foam sample with 10 wt% molybdenum.

Figure 4b. Scanning electron microscope photo of the same foam sample using back scattered electrons to illuminate the molybdenum.
CONCLUSIONS

• Molybdenum doped poly (4 methyl - 1- pentene) foams were successfully produced.

  • Doped TPX® foams with an estimated 25 wt% molybdenum were obtained.

  • TPX® doped foams with densities as low as 10.5 mg/cc were measured.

  • Current work shows that above 25 wt% Mo, the TPX® matrix may not be large enough and strong enough to support its own weight.

• The structure of the resulting foams are directly related to the cooling of the sample through the gelation temperature.

  • A gradient cooling system was designed and constructed to allow controlled heating, cooling and temperature monitoring of foam samples during processing.

  • The cooling rate through the gelation region has a direct effect on the final foam structure but more work is necessary to assess the effect on cell size and uniformity.
Now that doped foams have been successfully produced ..........

1. More sensitive measurement techniques must be used to determine the density, cell size, and dopant uniformity of each sample.

2. More thought must be given to the "real" requirements and restraints of the foam dimensions.

   What is the largest acceptable density (keeping in mind that the dopant adds to the density but not the strength of the foam)?

   How much dopant is necessary (remembering that too much dopant greatly compromises the low density and the strength of the resulting foams)?

   What are the cell size restrictions of an acceptable doped foam sample?

3. Optimization of the processing parameters to produce foams to meet "real" specifications is necessary.
IN SITU PRODUCTION OF VERY LOW DENSITY MICROPOROUS POLYMERIC FOAMS

Wigen Nazarov and John W. Falconer
University of Dundee, Department of Chemistry
Dundee DD1 4HN, Scotland

Colin J. Horsfield
Atomic Weapons Establishment, PLC.
Aldermaston, Reading RG7 4PR, England

Small, open ended parylene microcylinders of 400 \textmu m diameter, 700 \textmu m length and 10 \textmu m wall thickness were filled with a solution of a polyfunctional monomer of low concentration. The solution was polymerized in situ with U.V. light to produce a gel. Precipitation of these gels in a non-solvent and subsequent drying by means of a critical point drying apparatus produced microcylinders filled with a low density foam. Radiographic examination of the foam gave a density of the order of 2 \text{ mg cm}^{-3}, and SEM micrographs showed cell sizes of the order of 1 \textmu m.

No shrinkage of the foam was observed with cylinders of the dimensions given, but with larger cylinders of diameter and length or the order of 1000 \textmu m, some axial shrinkage was observed which resulted in cylinders with slightly concave ends (of the order of 20 \textmu m).
Introduction

This report details the production of low density foams of the order of 3 mg cm\(^{-3}\) and a pore size of approximately 1 \(\mu\text{m}\), for use in laser experiments. These foams were produced in cylinders of 400 \(\mu\text{m}\) diameter and 700 \(\mu\text{m}\) length.

Monomers, Solvent & Initiator

Low density foams were produced using the following monomers, solvent & initiator: Trimethylol Propane Triacrylate (TMPTA) or Penta Erythritol Tetraacrylate (PETA). Solvent: 1-decanol and Initiator: AIBN (2,2' Azobisisobutyronitrile). Three steps are involved in the in situ production of very low density foams:

1. Filling of microcylinders with a monomer solution and polymerization by photoinitiation

2. Precipitation (solvent exchange) of the gel and drying with critical point dryer

3. Characterization

1. Filling and Polymerization

A solution of the monomer in the solvent is prepared (0.3%), together with initiator (concentration of initiator is 1% of the monomer). The solution is then injected into a microcylinder (in this case a Parylene-N cylinder) which is then placed in a photolysis vessel (see figure 1). The vessel was then purged with nitrogen gas.

The monomer solution is then polymerized using a 200W U.V. light. At this point the monomer solution gels.

2. Precipitation of the gel and drying with Critical Point Dryer

The Cylinder containing the gel is placed in methanol, to exchange the solvent in the wet gel so that it can be exchanged with liquid CO\(_2\).

After the exchange the CO\(_2\) is vented and the dried cylinder containing the foam is taken out of the Critical Point Dryer.
3. Characterization

The low density foams produced were characterised by:
I. Radiography to measure the density and uniformity
II. Scanning electron microscopy to measure pore size and cell structure.

I. Radiography

The Foam filled cylinders were radiographed using a microfocus X-ray source - see figure 2. The system had:

A spot size of < 2 μm, a 6 μm thick Al source foil and was operated at an accelerated voltage of 6 kV. This produced a monochromatic source of X-rays at approximately 1.5 keV. Figure 3, is the radiograph of a foam filled cylinder shows a uniform although visually faint image. This figure also shows the image of a calibrating stepwedge which had 5 steps of 1.9 μm Parylene-N.

Figure 4, is the optical density plots of both the foam filled cylinder & the stepwedge show that the foam has produced a similar attenuation to that in 1.9 μm of Parylene-N. However, the scans have a very low signal to noise ratio, which limits the accuracy of this measurement to ± 1.0 μm of Parylene-N.

Density Measurement  The foam density is given by the following equation

$$C_c = \frac{C_{pyN}}{\mu_{pyK}} \cdot C_{pyN} \cdot X_{pyK} \cdot (\frac{\mu_{\tau}}{X_{\tau}})$$

Where:

- $C_c$ is the foam density
- $C_{pyN}$ is the density of Parylene-N in g/cc (1.12 g/cc)
- $\mu_{pyK}$ is the coefficient of absorption of Parylene N at 1.5 keV (527 cm² g⁻¹)
- $X_\tau$ is the foam thickness in μm (700 μm)
- $\mu_\tau$ is the coefficient of absorption of PETA at 1.5 keV (817 cm² g⁻¹)
- $X_{pyN}$ is the thickness of the stepwedge at the same optical density as the foam in μm (1.9 ± 1.0 μm).
Therefore the density of the PETA foam derived from the thickness of the stepwedge is:

\[ 2.0 \pm 1.0 \text{ mg cm}^{-3} \]

II Scanning Electron Microscopy

The surface of the very low density foams were characterised by SEM. The foams were coated with 750 Å of gold. Figure 5, this electron micrograph shows that the foam is composed of a network of submicron fibres and nodules. The pore size is predominately submicron, although there is a distribution of larger pores of up to 5 \( \mu \text{m} \). These, however, may be the areas of damage caused by the gold coating process.

Conclusions

In situ low density polymeric foams of the density in the order of 3 mg cm\(^{-3}\), and pore/cell size of 1 \( \mu \text{m} \), have been produced by the technique described here. These foams have been made in 400 \( \mu \text{m} \) diameter and 700 \( \mu \text{m} \) length cylinders. The larger diameter cylinders, however, have demonstrated some axial shrinkage.
The electron gun produces a beam of electrons which are focused into a 2 μm spot by the focusing coils. This spot impinges on a very thin metal foil and the x-rays are produced. The x-rays which pass through the foil are filtered such that they are predominantly characteristic of the foil energy of the x-ray. These x-rays then pass through the laser target and the x-ray shadow is projected onto the detector which is either an x-ray film or, for initial alignment, a phosphor coated intensifier optically coupled to a CCD (charge coupled device).

**Figure 1: Photolysis Vessel**

The radiograph of a foam filled cylinder shows a uniform although virtually faint image. This figure also shows the image of a calibrating stepwedge which had 5 steps of 1.9 μm Parylene-N.

**Figure 2: Microfocus X-ray Source**

**Figure 3:**
The optical density plots of both the foam filled cylinder (right) & the stepwedge (left) show that the foam has produced a similar attenuation to that in 1.9 μm of Parylene-N. However, the scans have a very low signal to noise ratio, which limits the accuracy of this measurement to ± 1.0 μm of Parylene-N.

II Scanning Electron Microscopy

The surface of the very low density foams were characterised by SEM. The foams were coated with 750 Å of gold.

This electron micrograph shows that the foam is composed of a network of submicron fibres and nodules. The pore size is predominantly submicron, although there is a distribution of larger pores of up to 5 μm. These, however, may be the areas of damage caused by the gold coating process.
Polymeric materials containing silicon are required for neutron diagnostics in the ICF program. Trimethylsilylstyrene polymers have been used by LLE. The synthesis of trimethylsilylstyrene will be described. In addition, using GA funds, GA has worked on making higher silicon content styrenes for polystyrene. We sought to make a styrene-CH₂CH₂-Si [Si(CH₃)₃]₃ monomer (I). Synthesis via the known Grignard route failed. However, hydrosilation of divinylbenzene with [(CH₃)₃Si]₃SiH, appears to form (I)

<table>
<thead>
<tr>
<th>material</th>
<th>SiO₂</th>
<th>(I)</th>
<th>C₈H₇-Si(CH₃)₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si atom%</td>
<td>33.3</td>
<td>6.56</td>
<td>3.57</td>
</tr>
</tbody>
</table>

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601 and GA Fusion IR&D.
Synthesis of Trimethylsilylstyrene Monomer

Grignard Synthesis Adds Trimethylsilyl to Styrene Readily

1) \(3 \text{CD}_3\text{MgI} + \text{C}_6\text{H}_5\text{SiCl}_3 \rightarrow \text{C}_6\text{H}_5\text{Si} (\text{CD}_3)_3 + 3 \text{MgICl}\)

2) \(\text{C}_6\text{H}_5\text{Si} (\text{CD}_3)_3 + \text{HCl} \rightarrow \text{Cl-Si} (\text{CD}_3)_3 + \text{C}_6\text{H}_6\)

3) \(\text{D}_2\text{C}==\text{CD}==\text{MgCl} + \text{Cl-Si} (\text{CD}_3)_3 \rightarrow \text{D}_2\text{C}==\text{CD}==\text{Si} (\text{CD}_3)_3\)

70% Yield of Monomer Upon Workup and Vacuum Distillation

Straightforward Polymerization to Form

Synthesis route previously used by Takagi at ILE Osaka

Efficient Synthesis of Tris(TrimethylSilyl)SilylStyrene Via a Grignard Reagent Fails

1) \([(\text{CH}_3)_3\text{Si}]_3\text{Si-H} + \text{CHCl}_3 \xrightarrow{\text{reflux}} [(\text{CH}_3)_3\text{Si}]_3\text{Si-Cl} + \text{CH}_2\text{Cl}_2\)

2) \([(\text{CH}_3)_3\text{Si}]_3\text{Si-Cl} + \text{H}_2\text{C}==\text{CH}==\text{MgCl} \xrightarrow{\text{THF}} 24 \text{hour workup} \xrightarrow{\text{H}_2\text{O}}\)

\(\text{H}_2\text{C}==\text{CH}==\text{Si}[\text{Si} (\text{CH}_3)_3]\) small amount (10%) made

large amounts of \(\text{H}_2\text{C}==\text{CH}==\text{Si}[\text{Si} (\text{CH}_3)_3]\) and \([(\text{CH}_3)_3\text{Si}]_3\text{Si-OH}\)

Grignard reaction step is very slow, or limited, due to bulk around the tris(trimethylsilyl)silyl group.

An alternate route to addition of TTS must be found-hydrosilation.
Hydrosilation of Divinylbenzene (DVB) With Tris(Trimethylsilyl)Silane (TTS) Works!

\[ \text{H}_2\text{C} = \text{CH} - \text{CH} = \text{CH}_2 + [(\text{CH}_3)_3\text{Si}]_3\text{Si}-\text{H} + \text{AIBN} \xrightarrow{80-90 \degree \text{C}} \text{C}_2\text{H}_4 \text{Si} - \text{Si} - \text{Si} (\text{CH}_3)_3 \rightarrow \text{uncrosslinked polymer} \]

1.0 mole DVB 1.3 mole TTS 0.25 mole

Hydrosilation reaction on styrene occurs more rapidly than polymerization of styrene. Apparently hydrosilation of divinylbenzene is more rapid than polymerization.

B. Kopping, et.al.,

Elemental Analysis of Si₄-Styrene Implies Copolymer

<table>
<thead>
<tr>
<th>Weight %</th>
<th>Carbon</th>
<th>Hydrogen</th>
<th>Nitrogen</th>
<th>Silicon</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₁₉H₃₈Si₄</td>
<td>60.3</td>
<td>10.05</td>
<td>0.00</td>
<td>29.68</td>
</tr>
<tr>
<td>Polymer made (exp)</td>
<td>60.7</td>
<td>10.04</td>
<td>0.85</td>
<td>24.63</td>
</tr>
<tr>
<td>Polymer shown below</td>
<td>63.5</td>
<td>9.85</td>
<td>0.89</td>
<td>25.76</td>
</tr>
</tbody>
</table>

- NMR shows no evidence of residual vinyl groups.
- Starting divinylbenzene was 20% ethylstyrene. Elemental analysis implies little incorporation of ethylstyrene.
- Nitrogen implies AIBN fragments in polymer. Amount of nitrogen is greater than just termination of tail of polymer chain.
- Ambiguity of chemical formula leads leads to ambiguity in Si atom%
Proton NMR of Si4-Styrene Shows Significant Si Content

\[(4 \text{ Si}/27 \text{ H}) \times 25.3 \text{ H (peak near 0.2 ppm)} = 3.75 \text{ Si for every 4 phenyl H (peak near 7.1 ppm)} \] corresponds to 6.1 Si atom%.

---

**Comparative Properties of Si4-Styrene With Other Shell Materials**

<table>
<thead>
<tr>
<th>Property</th>
<th>SiO2</th>
<th>Idealized C19H38Si4</th>
<th>Polymer made</th>
<th>Trimethylsilylstyrene C11H16Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si atom %</td>
<td>33.3</td>
<td>6.56</td>
<td>6.1 ±0.6 (NMR)</td>
<td>3.57</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>5.6 ±0.1</td>
<td></td>
</tr>
<tr>
<td>Density g/cc</td>
<td>2.65</td>
<td>~ 1.0</td>
<td>0.960 (gas pycnometer)</td>
<td>1.00</td>
</tr>
<tr>
<td>g Si/g material</td>
<td>0.4667</td>
<td>0.296</td>
<td>0.242 (x-ray fluores)</td>
<td>0.159</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.246 (element analysis)</td>
<td>0.159</td>
</tr>
<tr>
<td>g Si/cc of material</td>
<td>1.2368</td>
<td>0.296</td>
<td>0.2323</td>
<td>0.159</td>
</tr>
<tr>
<td>Refractive index</td>
<td>1.47</td>
<td></td>
<td>1.55</td>
<td></td>
</tr>
<tr>
<td>True ( M_W )</td>
<td></td>
<td></td>
<td>18.1 K</td>
<td>measured by combination</td>
</tr>
<tr>
<td>True ( M_N )</td>
<td></td>
<td></td>
<td>5.28 K</td>
<td>GPC, viscosity, and light</td>
</tr>
<tr>
<td>Polydispersity</td>
<td></td>
<td></td>
<td>3.43</td>
<td>scattering</td>
</tr>
<tr>
<td>Mark-Houwink Coefficients:</td>
<td></td>
<td></td>
<td>a = 0.286</td>
<td></td>
</tr>
<tr>
<td>intrinsic viscosity ( [\eta]_i = K M_a )</td>
<td></td>
<td></td>
<td>K = 0.003027</td>
<td></td>
</tr>
</tbody>
</table>
Si4-Styrene Shell Made by Microencapsulation

Polymer shell is mechanically weak-cracked

Shells typically had 3-6 micron walls

Si4-Styrene Shell Made in Drop Tower

Typical Wall Thickness 2-3 microns

Very Fragile Shells
Acknowledgements

Dr. Martin Hoppe measured the Si content of the polymer, and its refractive index.

David Husband made shells by the drop tower technique.

Dave Steinman and Don Beighley took pictures of the shells.

Micromeritics determined the polymer bulk density by gas pycnometry.

Viscotek measured the polymer molecular weight and related properties with their Triple Detector System (GPC, Light Scattering, and Viscosity Universal Calibration).

Galbraith Labs determined the elemental analysis.

Dr. Leroy Lafferty of San Diego State University obtained the NMR spectrum.

Tris(trimethylsilyl)silane was obtained from Fluka.

Divinylbenzene (80%) was obtained from Pfaltz & Bauer.

Synthesis of the trimethylsilylstyrene polymer was done under the DOE ICF contract.

Synthesis of the Si4-Styrene was done under General Atomics internal R&D funding.
AUTOMATION OF SELECTION AND CONTROL OF HOLLOW MICROSPHERES

Veselov A.V., Beinik A.Ja., Zakharov A.V., Inozemtsev A.P., Komleva G.V. and Punin V.T.

Russian Federal Nuclear Center, VNIIEF, Arzamas-16, Russia.

ABSTRACT

Methods for control of the shell diameter, wall thickness and wall nonuniformity and also methods for selection of shells in fluid have been developed and investigated. The methods allow us to separate shells according to the diameter with 0.25 μm accuracy, to the wall nonuniformity with 5-7% accuracy, to make measurements of the diameter with 0.35 μm accuracy and wall thickness with 3-10%, to rotate the shells with a small center beateng (0.1 - 1.0 μm).

These methods allowed us to develop the automated process cycle of control and selection of shells for laser fusion targets. A model automated line has been developed and joint operation of units has been investigated. The calculated automated line throughput during the shell analysis is 50-300 shells per hour, the control and selection accuracy for the diameter, thickness, and wall nonuniformity is 1%, 3-10% and 1-3%, respectively.

Thus the foundations have been laid for an automated, high-quality target production technology for power stations based on laser fusion.

INTRODUCTION

Microsphere parameter measurement is a labour-consuming process. It is practically impossible to draw a map of all the microsphere surface parameters during visual control. Automation for parameter control process and microsphere selection is required.

In all the works, known to us only separate stages of work are automated. In these systems microsphere rotation and results processing with the electronic computer are automated1-3. Microsphere selection, placing it on the manipulator and further operations, after measurements, performance, are carried out by hand. In the work1 the system for microsphere conveyance into the control zone is proposed, which is an electrostatic quadrupole line, where charged microspheres moves. But there is not any more information about such system creation and its efficiency.

METHODS OF CONTROL AND SELECTION AND ACCURACY OF CONTROL

By us automation methods of the control and selection in liquid were proposed and developed4-7.

The device for shells separation according to their diameter works in a wide range of diameters - from tens of microns to millimeters. It is capable of accomplishing both the rough separation of shells by batch in fractions with the diameters from tens to hundreds of microns, and the precision separation of the fractions with diameters in ranges of several tenths of a micron. The certificate of authorship for this device was received4. Photographs (Fig.1) show the separated shell fractions with diameter of 116.1±0.4 μm and 155.3±0.24 μm.

The device for separation of shells according to their wall nonuniformity is capable of selecting from a batch the shells with the wall nonuniformity better than 5-7%. The certificate authorship for the device was received5. Photographs (Fig.2.) show the shell fractions, separated according to their wall nonuniformity.

The other device allows us to determine the wall thickness with an accuracy of 0.1μm.

The next device is capable to determine the shell diameter with accuracy 0.35-1.0μm. The certificate authorship for this device was received6.

The devices for manipulation7 were developed also.
It was developed and investigated the several devices for rotation of sails with small beating (0.1 -1.0 μm). (Fig. 3). (Now they are patented). It was defined the optimal law of shell rotation for inspection of shell surface (Fig. 4).

The obvious advantages of this method are the following:
- the convenience of setting up and removal of a sphere in the arrangement;
- automatic setting up of the sphere into required position;
- increase of the rate of manipulation;
- little beating of the sphere center during its rotation (to 0.1 μm);
- broad access to the sphere surface for its control;
- any required law of the sphere rotation, determined by the laws of movement of supporting surfaces.

SCHEME OF AUTOMATIC LINE AND OPERATION

On the basis of those devices and methods a scheme and design of a line were developed. It allows us to automatize control and separation processes completely according to microsphere geometric parameters, qualitative microsphere selection, their manipulation and movement along the control devices. It is important, that all the operations are carried out in liquid and that is why all electrostatic charges on microspheres are eliminated, and their transference from device to device is secured by flotation along the guiding canals.

The scheme of the line is shown in Fig.5. The work of the line is accomplished in the following way. An initial microsphere batch (1) is sent into slit device (3), in which it is separated into fractions, according to diameters. The manipulator (8) conveys microspheres one by one, with an interval, into the device of preliminary separation according to nonuniformity (9). The commutator (6) transfers bad microspheres into the collector (7) and good ones to the interferometer (10). The interferometer carries out a precision measurement of nonuniformity with the help of the computer. Microspheres, which nonuniformity is more than the preset one, are transferred through the commutator (6) into the collector (7).

A microsphere with little nonuniformity floats into the device for wall thickness measurement (11). Then a microsphere gets to the corresponding device for diameter measurement (13). The computer determines microsphere wall thickness and diameter.

The microsphere gets into the cassette with numbered cells for keeping (14). In the computer memory there is information about the diameter, microsphere thickness and nonuniformity, kept in every cassette cell.

So, in the developed scheme not only automatic microsphere parameter measurement, but also microsphere transport to the measurement devices, setting and measurement sorting and moving of microspheres, which do not meet preset requirements, out of the line, and transference of standard microsphere into cassette for keeping are carried out. The most essential is the fact that all the operations, except for the initial loading of microspheres batch, are carried out without participation of an operator, and this allows work with any batch of microspheres, without preliminary microsphere selection.

The calculation efficiency of the proposed automatic line for microsphere analysis is 50-300 units per hour and depends on sphere diameters and preset control conditions. The output of standard microspheres will depend on the quantity of good microspheres in raw material. The control and selection accuracy for the diameter, thickness, and wall nonuniformity is 1%, 3-10% and 1-3%, respectively.

MODEL OF AUTOMATIC LINE

A model of automatic line was manufactured and assembled as the variant with manual control (without any computer) (Fig.6). Interaction of devices in the line structure for microsphere moving along all the route was developed. Microspheres move safely from device to device according to commutators commands. The further stage of development is devices equipment with an electronic control block and with a block for connection with the computer and development of all the arrangement.
FUTURE APPLICATIONS

As a matter of fact, an automated technological control cycle for microsphere selection for providing with targets of high quality for experimental investigations on the existed lasers has been developed and the basis of automated technology for the target fabrication for electric power stations employing ICF has been outlined.

Reference

a) $D = 116.1 \pm 0.4 \, \mu m$

b) $D = 155.3 \pm 0.24 \, \mu m$

**Fig. 1.** Fractions of shells, separated according to their diameter.

a) $\Delta d/d < 10\text{-}15\%$

b) $10\% < \Delta d/d < 40\%$

c) $\Delta d/d > 30\text{-}50\%$

**Fig. 2.** Fractions of shells, separated according to their wall nonuniformity.

**Fig. 3.** Devices for rotation of shells.

**Fig. 4.** The trajectory of control point and checked up the surface.
Fig 5. Scheme of the automatic line for selection and control of shells.
Fig. 6. Model of automatic line for control and selection of shells
HI-VOLTAGE ELECTRO-STATIC SHELL PICK-UP TOOL

D.H. Beighley, W.A. Baugh, and R.B. Stephens
General Atomics
P.O. Box 85608, San Diego, CA 92186-9748

Some ICF capsules such as large, thin walled plastic shells (i.e., 1000 μm O.D. x 5 μm wall thickness) and low-density foam shells, are so fragile that to move one with a vacuum chuck will destroy the shell. We are developing a hand-held, battery-operated, electro-static shell pick-up tool that will overcome this difficulty. This presentation will reveal the circuitry of the device we have built, its operating voltage and current load, and its effectiveness at handling a variety of shell types.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
The heart of a laser fusion target is the fuel capsule. Target specifications require the capsule to be of uniform wall thickness with excellent surface finish. Fuel capsules are currently fabricated by depositing polymer on a suitable mandrel using an RF excited plasma.

The surface roughness of plasma polymer coatings is dependent on the process conditions during the coating run. The effects of these process conditions, including feedstock flow rate, chamber pressure, coating thickness and temperature of the coating substrate, are examined. Atomic Force Microscopy (AFM) is used to measure the RMS surface roughness of the deposited polymer and the power spectrum subsequently calculated. Other physical properties of plasma polymer are examined and will be reported.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
The Effects of Process Conditions on Surface Roughness of Plasma Polymer Coatings

S.A. Letts, E.M. Fearon, G.W. Collins and M.D. Saculla
The plasma polymerization coating apparatus was modified to coat on a heated substrate.

Polymer coating quality is determined by the formation mechanism.

Gas feed mixture → Plasma → Dissociated, ionized products

Plasma formation → Etching

Particle nucleation → Surface mobility

Radical → Polymer coating

T_s → T_g → D_s
PROPERTIES OF CH PLASMA POLYMER

Gas feed
Trans-2-butane

\[
\begin{align*}
\text{CH}_3 & \quad \text{H} \\
\text{C} &= \text{C} \\
\text{H} & \quad \text{CH}_3
\end{align*}
\]

+ Hydrogen

Plasma

Polymer

\[
\begin{align*}
\text{CH}_3 & \quad \text{CH}_3 & \quad \text{H} & \quad \text{CH}_2 \\
\text{C} = & \quad \text{C} & \quad \text{C} = & \quad \text{C} & \quad \text{C} = & \quad \text{C} \\
\text{CH}_3 & \quad \text{CH}_2 \\
\text{H} & \quad \text{H} & \quad \text{CH}_3
\end{align*}
\]

Atomic composition: $\text{CH}_x, 1.2 < x < 1.4$

Density: 1.0 g/cm$^3$

Refractive index: 1.6

Color: Yellow (blue absorbing)

Changing flow conditions produce sub 100 Å surface finish for plasma polymer coatings

Trans-2-butene flow (sccm)

0.28

$\sigma =$ 89.5 nm

0.17

$\sigma =$ 14.7 nm

Hydrogen flow (sccm)

11.6

$\sigma =$ 0.9 nm

23.1

$\sigma =$ 0.5 nm

35.6
The scaling of surface roughness changes with T2B flow

Thicker coatings at constant coating conditions result in longer wavelength surface roughness

- .014 μm thick: 2.9 nm RMS
- 1.1 μm thick: 15.2 nm RMS
- 8.5 μm thick: 45.5 nm RMS
The power spectrum shows the RMS surface finish increasing with film thickness.

Plasma polymer surface roughness increases with increasing deposition rate.

10 µm thick films

- 27.3 µm thick film: 1.5 µm/hr rate, 42.4 nm RMS
- 15.5 µm thick film: 1.2 µm/hr rate, 12.6 nm RMS
- 6.2 µm thick film: 1.0 µm/hr rate, 1.2 nm RMS

0.23 sccm T2B
11.6 sccm H₂
12 watts
72 mTorr
The surface roughness of plasma polymer decreases with increasing substrate temperature.

54°C
39.9 nm RMS

75°C
15.4 nm RMS

88°C
3.7 nm RMS

Surface finish decreases with increasing temperature.
Increasing the substrate temperature greatly reduces RMS roughness and slightly reduces deposition rate.

![Graph showing RMS roughness and deposition rate as functions of 1000/T(Si), K^-1.]

Closed symbols 5 sccm H₂, Open symbols 10 sccm H₂.

Conclusions - plasma polymer surface finish analysis

- Surface finish can be controlled through process conditions
- RMS roughness, \( \sigma \), increases with thickness:
  - \( \sigma \sim t^{0.7}, \ R = 2\mu m/hr \)
  - \( \sigma \sim t^{1.0}, \ R = 1\mu m/hr \)
- Roughness increases with rate:
  - \( \sigma / \text{thickness}^3 = 0.4R^{4.46} \)
  - Low rate allows surface diffusion to smooth surface
- Roughness is a function of temperature:
  - For \( T < 80^\circ C \) \( \sigma / 0.4R^{4.46} \text{thickness}^3 \approx \text{Constant} \)
  - For \( T > 80^\circ C \) \( \sigma / 0.4R^{4.46} \text{thickness}^3 = \exp(13,213/T) \)
  - Increased surface diffusion allows filling of valleys
- Depositing at a rate of 0.8 \( \mu m/hr \), coatings 50\( \mu m \) thick are produced with a 2 nm RMS surface roughness
- Roughness scaling provides a tool for further investigation of the mechanism of plasma polymerization
An understanding of the fundamental mechanisms underlying plasma polymerization, especially in connection with surface roughness evolution, would increase the applicability of this technology to advanced Inertial Confinement Fusion (ICF) target fabrication. Chemical reaction kinetics and surface diffusion modeling to describe film growth and roughness is aided by characterization of the gas phase coating environment with respect to the identity of species and their concentrations at the point of interaction with the substrate. Mass spectrometry is being used in this laboratory to characterize the plasma polymerization coating environment.

Evidence shows that coater deposits come from both the conversion of organic feed gas and plasma etching of polymer deposited in the silica plasma envelope. Mass spectral evidence will be presented to demonstrate this fact. The etching reaction favors the creation of lighter fragments to heavier ones. It is hypothesized that the etching reaction channels energy into the growing film interface and may play a role in determining the evolution of surface roughness via surface diffusion. A presentation of evidence for this and the correlation between the gas phase composition and surface roughness will be presented.

If deuterium is substituted for hydrogen during plasma polymer etching, it should be possible to ascertain the level of deuterium exchange with the surface and/or the liberated organic species. Any hypotheses regarding etch reaction pathways must be consistent with these data. Similar work with $^{13}$C is also considered.
Mass Spectrometric Determination of the Coating Environment in Plasma Polymerization Under Rough and Smooth Coating Conditions

R.M. Brusasco, G. Wilemski, S. Letts and R. Cook

Target Science and Technology

University of California
Lawrence Livermore National Laboratory

Target Specialist Meeting
Taos, NM
February 6-9, 1995
A greater understanding of the film growth mechanism is needed for optimization of the coating process, particularly in the evolution of surface roughness.

*The goal is to obtain higher deposition rates (X 2-3) with surface roughness $\leq 100 \, \text{Å rms}$. *

Practical application begins by characterizing the gas phase growth environment and investigating changes in that environment under various process conditions.

A quadrupole mass spectrometer is close-coupled to the helical resonator plasma applicator.
Growth units adsorb, diffuse and bond to build the polymer surface.

Growth unit "G" could be neutrals, radicals or ions and either hydrocarbons or even hydrogen. Hydrogen abstraction leads to active surface sites.

Radical sites occupy the surface since their concentration in the bulk is quite high.

Growth units diffuse until a suitable site is found, where bonding occurs.

Electron impact and plasma decomposition produce different fragmentation patterns

Differing partial pressures account for the different ion currents
Gas phase oligomers are clearly evident.

T2B Parent Ion = 56 amu

Therefore, growth units could comprise hydrocarbon species ranging from C1 to possibly C7 in size.

Normalization to the total hydrocarbon ion current provides a process "fingerprint."

A fully calibrated mass spectrometer determines partial pressures via "relative sensitivity factors" $a_i$:

$$\sum_i a_i I_i = \sum_i p_i = P_{total}$$

The current $I_i$ for species $i$ is also a function of the ionization process in the spectrometer. To produce a process "fingerprint" one normalizes a hydrocarbon fragment peak against the total hydrocarbon ion current:

$$\zeta_i = \frac{I_i}{I_{total}}$$

The hydrogen partial pressure is large and can be neglected to first order.
The fragment distribution under smooth surface deposition conditions is dominated by light masses.

Increasing precursor flow rate speeds deposition but roughens the surface as well.

Roughness scaling with thickness

How does one move towards the origin of this plot?
Increasing precursor flow rate shifts the fragment distribution towards higher masses.

The trend suggests a shift towards a larger, heavier average growth unit.

Hydrocarbon fragments are detected even with zero precursor flow.

Indicates that etching of polymer deposited upstream is occurring and that the gas phase is composed of homogeneous and heterogeneous decomposition products.
A reduction in operating frequency has been found to speed deposition without roughness penalty.

Frequency reduction shifts the fragment distribution towards smaller species.

Constant gas flow rates and resonator power dissipation.
The effects seen with a change in RF drive frequency is thought to work via a change in the spatial distribution of power within the plasma envelope. Lowering the frequency should increase the power dissipation nearer to the substrate.* This results in enhanced energy transport to the growing interface, as indicated by higher substrate temperatures, and thus increases surface diffusion (smoother coatings).

For otherwise constant conditions, the partial pressures of hydrocarbon fragments increases at lower operating frequency. This fact explains the increased coating rate.

---


Mass spectrometry is aiding our understanding of plasma polymerization.

- The close-coupled mass spectrometer reveals trends in the gas phase composition which are correlated with improvements in the deposition rate/surface roughness behavior.

- Increased precursor concentration alters the fragment distribution, favoring heavier fragments over lighter ones. This is correlated with rougher surfaces.

- A reduction in the operating frequency increases the production of lighter fragments relative to heavier ones and is correlated with higher deposition rates and smoother surfaces. The role of etching in roughness development needs further investigation.

- Calibration to obtain partial pressures of species is urgently needed.
Acknowledgments

Anselmo Dueñas
Evelyn Fearon
Gilbert Collins
Michael Saculla

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
The target insertion system for OMEGA Upgrade must meet exacting and sometimes conflicting design requirements. The original cryogenic elevator concept has undergone several revisions in response to analysis and comparison of projected performance with the design requirements. The implicit requirement that the cryogenic shroud be removed a minimum of 20 cm from the target in the last 20 ms before the shot has a significant impact upon the design. The updated cryogenic elevator concept will be presented along with insertable cryostat and augmented mass free fall target insertion concepts.

*Work supported by U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
HIGH PRESSURE TARGET FILL SYSTEM FOR THE OMEGA UPGRADE ICF LASER*

R.L. Fagaly, N.B. Alexander, R.A. Mangano, and C.R. Gibson
Fusion Group, General Atomics
P.O. Box 85608, San Diego, CA 92186-9784

The OMEGA Upgrade ICF laser will use millimeter-size targets filled with D₂ or DT fuel and maintained at cryogenic temperatures. This requires a system used for diffusion filling of ICF targets to K bar pressures, cooling (below the triple point), and removal from the permeation cryostat, while maintaining a "zero" tritium release (to the environment) limit.

We describe the current status of the fill station and the development of equipment designed to be DT compatible, function at high pressure, and optimized for cryogenic temperatures. Particular emphasis will be paid to the permeation cryostat and a unique cold transfer cryostat used to transport fill targets at cryogenic temperatures.

*Work supported by the U.S. Department of Energy under Contract No. DE-AC03-91SF18601.
The OMEGA Upgrade requires millimeter-size Inertial Confinement Fusion (ICF) targets filled with D₂ or DT fuel and maintained at cryogenic temperatures. The fuel will be layered on the inner surface to a thickness as great as 100 μm. This dictates that empty shells be filled to densities (at room temperature) as high as 0.16 gm/cc; equivalent to a pressure greater than 1100 atm, and then cooled to cryogenic temperature. This mandates the development of a high-pressure, high accuracy, cryogenic pressure transducer to accurately assess the amount of D₂ or DT fuel in the target. A Paroscientific pressure transducer was used as the baseline. It is a high pressure (0–20,000 psia), high accuracy (0.001% of F.S.) gauge that measures changes in the resonant frequency of a piezoelectric quartz crystal. We will discuss the modifications to this unit, in particular the development of low temperature electronics, and the experiments used to validate the prototype pressure transducer.
LOW TEMPERATURE PROPERTIES OF THE POLYSTYRENE CAPSULES
MADE BY SWELLING TECHNIQUE

E.R.KORESHEVA, I.E.OSIPOV, I.V.ALEKSANDROVA
P.N.LEBEDEV INST., LENINSKIY PR.53, MOSCOW 117924, RUSSIA.

The typical target for the experiments on power laser facility consists of a polystyrene capsule filled with D-D or DT fuel up to high pressure ($P \leq 1543$ atm). The corresponding thickness of fuel layer inside cryotarget ranges from 10$\mu$m up to 180$\mu$m. One of a key problem of a future inertial confinement fusion (ICF) experiments is: "How to fill polystyrene capsule with fuel up to high pressure and transport it to the layering module without capsule destruction and fuel loss?".

To consider this question we have inspected the polystyrene capsules created at P.N.Lebedev Physical Institute by swelling technique (PST-capsules). As a result it is found that factor of strength of PST capsule increases while factor of hydrogen permeability and inner fuel pressure decreases under capsule cooling. The results are presented. It has been estimated that filled capsule can be transported to the layering mobile or directly to ICF chamber at temperatures more than critical one if capsule aspect ratio doesn't exceed the value 50 and filling pressure doesn't exceed 1500 atm. Such data allow one to use in ICF experiments cold gas-filled targets, one phase liquid and critical one (filling pressure equal or more than 580 atm for D-D fuel and than 600 atm for D-T) as well as to apply a technique of inverse FIF). We have created apparatus and carried out the experiments with cryolayer formation inside PST capsules using FIF. As a result of this study a system to fill PST capsules up to high pressure has been conceptually designed.
REFERENCES


PERMEABILITY FACTOR STUDYING

The created equipment and the developed interferometric method allow us to measure the factor of permeability of the wall of polystyrene capsule for deuterium at different temperatures.

CRITICAL PRESSURE $P_{cr}$ AND CHARACTERISTIC TIME $\tau$ OF THE POLYSTYRENE CAPSULE IN DEPENDENCE ON THE ASPECT RATIO $A_s$

$$P_{cr} = \frac{2E}{A_s}^{\frac{2}{3}} \left(1 - \nu^2\right)^{1/2}$$

$$\tau = \frac{R^2}{3F \cdot A_s \cdot P_H}$$

($E = 10^4 \text{kg/cm}^2$, $\nu = 0.3$)

$$R \gg \Delta R$$

<table>
<thead>
<tr>
<th>$A_s$</th>
<th>$P_{cr, \text{atm}}$</th>
<th>$\tau$, min (300K)</th>
<th>$\tau$, month (140K)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$R=500 \mu m$</td>
<td>$R=1000 \mu m$</td>
<td>$R=500 \mu m$</td>
</tr>
<tr>
<td>10</td>
<td>120</td>
<td>16.3</td>
<td>65</td>
</tr>
<tr>
<td>20</td>
<td>30</td>
<td>8.2</td>
<td>32.5</td>
</tr>
<tr>
<td>35</td>
<td>9.8</td>
<td>4.7</td>
<td>18.6</td>
</tr>
<tr>
<td>50</td>
<td>4.8</td>
<td>3.3</td>
<td>13</td>
</tr>
</tbody>
</table>

$T = 300K$ - FILLING

$T < 140K$ - $D_2$-TRAP TEMPERATURE
FIG. 2  FILLING CHAMBER DEPRESSURISING AND TARGET TRANSPORT TO
THE LAYERING MODULE
POLYSTYRENE CAPSULES MADE IN LPI (SWELLING TECHNIQUE)

Measurements of the tensile strength limit of a capsule

\[ 2R = 350-800 \, \mu m \]
\[ \Delta R = 5-12 \, \mu m \]
\[ A_s = 15-85 \]
\[ \tau = \frac{2\sigma}{A_s} \quad A_s \gg 1 \]

OUR EXPERIMENT

300K \( \sigma = 180-390 \) kg/cm\(^2\)
108K \( \sigma = 580-704 \) kg/cm\(^2\)

EXPECTED IN OPTIMAL REGIME

300K \( \sigma = 300-350 \) kg/cm\(^2\)
100K \( \sigma = 3.5\sigma_0 = 1050-1225 \) kg/cm\(^2\)

THE CHOICE OF THE TEMPERATURE OF GAS RELEASE AND TARGET TRANSPORT HAS TO BE DONE AT ANY PARTICULARLY CASE.

\[ \sigma_1 = 1140 \text{ kg/cm}^2 \quad \sigma_2 = 700 \text{ kg/cm}^2 \]
Table 1 THE PARTICULAR EXAMPLES

\( E_L = 30k\text{J}, D_2 - \text{FUEL, POLYSTYRENE CAPSULE, } \sigma = 700\text{kg/cm}^2 \) at. \( T \leq 100K \)

<table>
<thead>
<tr>
<th>I. INPUT DATA</th>
<th>( N\text{_1} )</th>
<th>( N\text{_2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inner radius, ( R_0 )</td>
<td>500 ( \mu\text{m} )</td>
<td>340 ( \mu\text{m} )</td>
</tr>
<tr>
<td>Aspect ratio, ( \text{As} )</td>
<td>50</td>
<td>35</td>
</tr>
<tr>
<td>Cryolayer thickness ( W )</td>
<td>100 ( \mu\text{m} )</td>
<td>30 ( \mu\text{m} )</td>
</tr>
<tr>
<td>at ( T = T_{lr} ) (solid)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>II. ESTIMATED DATA</th>
<th>( N\text{_1} )</th>
<th>( N\text{_2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filling pressure, ( P_f )</td>
<td>980 atm</td>
<td>360 atm</td>
</tr>
<tr>
<td>Filling density, ( \rho_f )</td>
<td>96.2 mg/ccm</td>
<td>47.9 mg/ccm</td>
</tr>
<tr>
<td>Parameter ( \Theta )</td>
<td>1.38</td>
<td>0.69</td>
</tr>
<tr>
<td>Time of filling, ( t_f )</td>
<td>( \sim 16 \text{ hours} )</td>
<td>( \sim 2 \text{ hour} )</td>
</tr>
<tr>
<td>Temperature ( T_S ) of gas-liquid separation</td>
<td>37.6 K</td>
<td>37.8 K</td>
</tr>
<tr>
<td>Cryolayer thickness</td>
<td>500 ( \mu\text{m} )</td>
<td>0</td>
</tr>
<tr>
<td>at ( T = T_S )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature ( T_r ) of depressurizing (upper limit)</td>
<td>41 K</td>
<td>57 K</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>III. FUEL STATE INSIDE THE CAPSULE</th>
<th>( N\text{_1} )</th>
<th>( N\text{_2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>One-phase state:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gas</td>
<td>( 38.34K &lt; T &lt; 41K )</td>
<td>( 37.8K &lt; T &lt; 57K )</td>
</tr>
<tr>
<td>Liquid</td>
<td>( 37.6K &lt; T &lt; 38.34K )</td>
<td>-</td>
</tr>
<tr>
<td>Two-phase state:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Liquid cryolayer</td>
<td>( 18.7K &lt; T &lt; 37.6K )</td>
<td>( 18.7K &lt; T &lt; 37.8K )</td>
</tr>
<tr>
<td>Solid cryolayer</td>
<td>( T &lt; 18.7K )</td>
<td>( T &lt; 18.7K )</td>
</tr>
</tbody>
</table>
The key to using the polymer or glass capsules for cryotarget fabrication with thick layers lies in the development of a special system to fill the capsules with gas fuel up to the high pressure.

1. THE CONCEPT OF THE FILLING CASSETTE

- Basic concept: filling chamber is cooled by liquid helium flow.

Filling temperature: $T = 300 \text{ K}$

$D_2$-trap temperature: $T < 140 \text{ K}$

Storage temperature: $T > 40 \text{ K}$

POLYSTERENE CAPSULES

(SWELLING TECHNIQUE)

Operation procedure:

1. Gas collection from accumulator into cryoconcentrator; $T = 4.2 \text{ K}$
2. Filling (step by step); $T > 300 \text{ K}$
3. Gas collection from filling line into cryopump; $T = 4.2 \text{ K}$
4. Gas return into accumulator
The physics of fuel layer formation with various internal structures has been studied in regard to the development of promising layering techniques of ICF experiments with laser energy greater than 30 kJ. The phase dynamic of fuel under gas-filled capsule cooling is found to depend on whether the initial gas density is more, less, or equal to the critical density for a given fuel material. The regularities found agree closely with those obtained from the experiments with high-strength glass capsules filled with hydrogens up to 1200 atm at 300 K. Since the choice of a formation isochor dictates the temperature dependence of the relative cryotarget parameters, it allows us to determine the initial gas density at fuel filling that is the initial fill pressure with non-destructive testing. The temperature life-area of different target types (namely gas-filled, liquid, critical and two-phase) with polystyrene capsules made by swelling technique (tensile strength is about 350 kg/scm at 300 K) is calculated for initial fill pressure in the range of 70-1543 atm.

Note that only two-phase targets call for special-purpose methods of cryolayer formation. Along with the studies of thick quasi-amorphous and liquid layers, a new technique to homogenize a polycrystalline cryolayer has been proposed and examined. The operational temperature was 4.2 K. The minimal time to achieve the complete homogeneity of solid hydrogen was 150 sec. The temperature range of stable existence is up to the triple point temperature. Such a volumetric effect can be caused by orientational ordering of nuclei spins which is accompanied by the transition between hcp and fcc structures and may result in a single crystal layer formation. The changes in the morphology of the frozen hydrogen isotope layers under the target heating have been investigated experimentally for each cryolayer structure obtained.
Techniques used to create thick liquid layers in capsules with fill tubes will be presented. A system of heat switches and a control volume provides a means to introduce liquid into a capsule in a controlled manner. This procedure and also the method we use to create thermal gradients will be discussed in detail.
Millimeter size capsules with attached fill tubes are currently being used for liquid layering experiments. These capsules are free standing plasma polymer shells made from coated poly(a-methylstyrene) mandrels. The fabrication process requires drilling a 150μm hole, leaching away of the solid mandrel, characterizing the capsule, and attaching a fill tube. Fabrication techniques and processes will be presented in detail.
Fabrication Techniques for Cryogenic Targets

TENTH TARGET FABRICATION SPECIALISTS MEETING
February 6-10, 1995

John Burmann*
Diana Schroen-Carey*
Steve Letts**
Jorge Sanchez**
*W.J. Schafer Assoc.
303 Lindbergh Ave. Livermore, CA 94550
(510) 447-0555
**Lawrence Livermore National Laboratory, Livermore, CA 94550
(510) 422-8381
These are the uncoated poly(α-methylstyrene) mandrels.

The mandrel sizes are currently 1 and 2 mm. diameter.

These mandrels have been plasma polymer coated to a wall thickness @ 40-80 μm.
The coated mandrels are then secured to a glass slide with PVA (polyvinyl-alcohol) and a hole drilled to 150 µm dia.

The mandrel is then dissolved by injecting MEK into the drilled hole until it is clean.
The shells are then characterized using interferometry or microscope inspection.

The shell is then attached to a 150 μm dia. glass fill tube using a cryogenic epoxy (Crest # 212).
* The shell with attached fill tube is checked by interferometry to insure an open fill line by watching for a fringe change as it is being pumped out.

* The shell is then vacuum leak tested at room temperature and at LN₂ temperature.

Here is a shell mounted in the Square Cell used for liquid layering experiments at LLNL.
This is a shell mounted on the Clamped Shell mount also used for liquid layering.
The goal of liquid layering experiments for mixed-fuel IFE targets is to use thermal gradients in surface tension to create a uniform liquid layer around a centered vapor bubble within a hollow solid capsule. Control of thermal profiles is critical to establishing and maintaining liquid layers with the desired shape. This study used thermal models for the liquid layering experiments to examine the effect of variations in the laser heating profile on thermal profiles within the capsule.

A finite element differential equation solver (PDEase2) was used to model thermal transport in a variety of experimental geometries. A thermal conduction model was used with different effective conductivities in the vapor, liquid and solid sections of the capsule. Calculations were done for the fixed-plate experiment where the bottom of the capsule was assumed fixed at 20 K and for the helium-background experiment where the target is supported on a long thin stalk within a helium gas. In all cases, volumetric heating of the solid shell by a laser was assumed. For each case variations were done on total absorbed laser power and the shape of the axisymmetric heating profile. The thermal profiles and the maximum temperature rise within the capsule have been used to explain the behavior observed in the fixed-plate experiment and to predict the laser powers and profiles which will be needed to create a uniform layer within the helium-background experiment.

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Thermal Modeling of Liquid Layering Experiment

Dr. Robert Bieri
W. J. Schafer Associates, Inc.

Target Fabrication Specialist Meeting
Taos, NM
February, 1995
Objectives of Work

The principal objectives of this study are:

To examine the effects of changes in the effective conductivity of the liquid and vapor (due to changing convection rates) on internal capsule temperature profiles.

To examine the effect of changes in the applied external temperature or heating profiles on internal temperature profiles.

To predict external heating or temperature profiles needed to create a uniform (cos(θ), where θ is the angle made with the z axis) temperature gradient (and thus a uniform surface tension gradient) at the liquid/vapor surface.

To understand an oscillatory mechanism which may result from temperature fluctuations.

The goal has been to apply a constant dT/dz to the shell. The fluid can only respond to the dT/dθ on the inside of the shell. The presence and motion of the fluid alters the temperature distribution, so the challenge is to find the proper heat distribution to apply to the capsule to create the desired dT/dθ on the inner liquid surface.
Description of Capsule Model and Preliminary Case Studies

Because PDEase® cannot solve complicated convection problems, all capsule regions are modeled as solids and thermal profiles are generated by solving the conduction equation:

\[ \nabla \cdot (k \nabla T) = S \]

where:
- \( k \) is the thermal conductivity of the solid (W/m/K),
- \( T \) is the temperature (K), and
- \( S \) is the volumetric heating rate (W/m\(^3\)).

Regions of the capsule can be established with various values (constant or profile functions) for \( k \) and \( S \). Boundary conditions at each region’s surface can be set for either the value or the normal derivative of the temperature. The temperature and heat flux distributions are then solved for throughout the capsule.

The capsule dimensions are taken from those of the experiment (2 mm diameter capsule with 44 micron wall thickness and a 0.845 mm post diameter). To allow better viewing of the temperature behavior in the shell, the wall thickness and the effective conductivity are both multiplied by 5. This leaves the heat flux through the shell invariant.
Case Descriptions

The first study examines the effect of changes in effective conductivity. As a liquid layer is established, convective heat flow will transfer heat more efficiently than the stagnant vapor. Once evaporation and condensation begin, the heat transfer can be $10^5$ times that of the vapor. The first set of calculations examines an unheated target between two fixed temperature plates (at 20 and 30 K). The 2 region model of the capsule is used, and changes in the thermal gradient within the capsule and the power flow through the capsule with changes in the effective conductivity of the central region are examined.

For the second set of calculations, a target between two fixed temperature plates (both held at 20 K) is heated with a laser. Thermal profiles within the shell are examined for a variety of heating profiles within the outer shell. These profiles are compared with the desired thermal profile consistent with a specified $\cos(\theta)$ temperature distribution on the outer capsule surface. The heating profiles examined are:

I. A volumetric heating rate in the upper shell $\sim \cos(\theta)$
II. A very steep (spot) heating rate $\sim \exp(-A \theta^2)$
III. A volumetric heating rate in the upper shell $\sim \sin(\theta)$
IV. A volumetric heating rate in the upper shell $\sim \sin(2 \theta)$

Results of Changing Heat Flow Efficiency
Results of Changing Heat Flow Efficiency

Results of Two Region Model - Fixed Cos(θ) External Temperature
Results of Two Region Model - \( \cos(\Theta) \) Volumetric Heating of Shell

1 Milliwatt \( \cos(\theta) \) Heating (10x conductivity in center)

Results of Two Region Model - \( \exp(-30.516* \Theta^2) \) Volumetric Heating of Shell

1 Milliwatt \( \exp(-\theta^2) \) Heating (10x conductivity in center)
Results of Two Region Model - Sin(θ) Volumetric Heating of Shell

1 Milliwatt Sin(θ) Heating (10x conductivity in center)

Temperature Rise
Vol=2.886E-6
max 1.6400
min 0.000
r = 1.6400
a = 1.5500
q = 1.0000
n = 1.4000
m = 1.3500
x = 0.2200
l = 0.2000
i = 0.1500
= 0.1000
f = 0.0000
θ = 0.7500
a = 0.8500
b = 0.8000
min = 0.7020

Results of Two Region Model - Sin(2θ) Volumetric Heating of Shell

1 Milliwatt Sin(2θ) Heating (10x conductivity in center)

Temperature Rise
Vol=2.533E-6
max 1.5410
min 0.000
r = 1.5410
a = 1.5000
q = 1.4000
n = 1.2500
m = 1.2000
x = 0.1000
l = 0.0500
i = 0.0000
= 0.0000
f = 0.8500
θ = 0.8000
a = 0.7000
b = 0.6500
a = 0.6000
min = 0.5980
The following power distributions all deposit ~ 1 mW total power in the top half of a spherical shell. The angle made with the vertical (z) axis is given by θ.

\[ P_1(\theta) = \frac{P_{\text{tot}} \cdot \cos(\theta)}{\pi \cdot r^2 \cdot t_{\text{shell}}} \]

\[ P_2(\theta) = \frac{P_{\text{tot}} \cdot \sin(\theta)}{\pi \cdot r^2 \cdot t_{\text{shell}}} \]

\[ P_3(\theta) = \frac{P_{\text{tot}} \cdot \sin(\theta)}{\frac{4 \cdot \pi \cdot r^2}{3}} \]

\[ P_4(\theta) = \frac{P_{\text{tot}} \cdot \sin(\theta)}{\frac{2 \cdot \pi \cdot r^2}{3}} \]

### Comparison of 4 Volumetric Heating Profiles

1. Uniform (radial) heating (no conductivity in center)
2. Uniform (axial) heating (no conductivity in center)
3. Uniform (radial) heating (no conductivity in center)
4. Uniform (axial) heating (no conductivity in center)
Relevance of Results to Experiments - Layer Shaping

The temperature profile along the inner shell surface will approximate the shape of the temperature profile along in liquid/vapor surface. By examining the spacing of the isotherms in the PDEase output, we can see the relative strengths of the surface tension gradient in various parts in the capsule. You would expect the experiment to have thin spots in the liquid layer, wherever this gradient is relatively low. For example, the output below (for a cos(Θ) heating profile) shows a temperature profile consistent with a thick layer on top of the capsule which thins as you approach the capsule equator.

Layer Shaping - Continued

Both the cos(Θ) and the sharply peaked exponential heating cases give profiles which have low thermal gradients as you approach the equator. It may be possible to super-impose an annular heating source with a source centered near the top of the target to approximate the uniform temperature gradient given by specifying an external cos(Θ) temperature distribution. An appropriately scaled combination of the Cos(Θ) and Sin(Θ) or Sin(2 Θ) profiles shown below could approximate the desired distribution.
Oscillations

Unfortunately, the effective conductivity of the vapor and the liquid are not steady-state quantities. In fact, as layer thickness changes and evaporation/condensation begins, the effective conductivity of both the liquid and the vapor can change by large factors in a very short time. You can imagine an oscillation produced as follows:

- A large initial temperature gradient is created inside the capsule because there is no liquid layer.
- The liquid is rapidly pulled up by the surface tension gradient.
- This moving liquid convects heat and reduces the temperature drop across the shell.
- The lower temperature gradient cannot support the raised liquid which drains down.
- The small remaining liquid layer convects less heat and leads to a larger temperature gradient.
- The larger temperature gradient raises more liquid and the cycle repeats.

This can all take place with constant power flow through the target.

Conclusions

Although we do not yet have a tool which will let us properly examine the time-dependent fluid behavior in a capsule, we can gain some important insights into the experimental behavior using a simple conductive solid model. This model has been used to study the effects of changes in effective conductivity of the liquid and vapor on temperature distributions inside the capsule and to study the temperature distribution created by various combinations of heating profiles and assumed effective conductivity. Basic conclusions of this work are:

The effective conductivity of the fluid in a capsule has a significant effect on the temperature distribution throughout the shell.

Temperature and heat-flux distributions may have fundamentally different shapes than applied volumetric heating distributions.

The desired laser heating distribution must be calculated from:

- The estimated effective conductivity of the fluid layer and vapor bubble
- The desired temperature distribution at the liquid/vapor surface (assumed $\sim \cos(\Theta)$ here and in Model I)

Rapidly changing effective conductivity (due to convection and phase transitions) can provide an oscillation mechanism which has not yet been examined.
Conclusions

The desired heating profile for an internal temperature gradient varying linearly with $z$ will have significant heating far from the capsule center; a $\sin(2\Theta)$ heating distribution is much closer than the $\cos(\Theta)$ distribution first calculated. This should solve the problem of layer thinning away from the poles.

It is crucial that temperature fluctuations be avoided as the liquid layer is formed as this can provide a powerful driver for fluid oscillations. We guess that the best experimental procedure will be to increase the laser power in small increments with a delay time between power increases long enough to allow temperature oscillations to die down.
Kinetics of freezing of microtargets filled with gaseous deuterium and deuterium-tritium mixture is investigated. A numerical simulation of the D-T mixture condensation process within a closed spherical balloon has been performed. A cryogenicspherical layer of the D-T mixture with the thickness inhomogeneity less than 20% has been obtained in experiments by the rapid freezing method. During rapid freezing of a target we observed nucleation in the microspheres volume. It is shown that with this method a homogeneous cryogenic layer can be obtained only in small diameter microspheres.

We have performed an investigation of the liquid D-T mixture behavior in spherical targets. It is shown theoretically that a homogeneous spherical layer of one-component fluids can be obtained by providing the temperature distribution over the external surface of a target $T=\text{A}\times\cos\theta$ ($\text{A}$ is the constant, $\theta$ is the angle measured from the vertical axis). Calculations have been performed aimed to find the temperature distribution in container walls for providing of this temperature distribution at the target’s surface. The liquid D-T mixture distribution has been investigated in a spherical target connected with a cold thermostat by a metallic wire in different temperature environments.

The cryogenic layer destruction has been investigated in a chamber with walls under the room temperature.

The active model of the system is described that allows to fabricate cryogenic targets directly in the laser chamber.
We present results of investigations of the freezing process for spherical microtargets filled with gaseous deuterium or deuterium-tritium mixture. Moreover, we have investigated the behavior of liquid and solid layers of these isotopes. The work has been performed mainly in the time interval from 1980 to 1990 years.

1. The experimental investigation of the freezing kinetics was performed according to scheme shown in Fig. 1. A gas filled glass target (1) was placed in an isolated chamber (5) connected through a thermal bridge (3) with the cooled part of a helium cryostat. The target was hanged to the top of chamber by a glass or metal fiber (2). The chamber was surrounded by a shield that had the temperature of liquid nitrogen (6) and by the walls at room temperature (7). The room inside cryostat was evacuated. The target was viewed through windows (4). A thermal exchange gas (helium or hydrogen) filled the chamber under pressure in the range of boundaries 0.001-1000 Pa. The chamber temperature could be varied from 9 to 30 K. The CW laser (8) illuminated the target during observation of the fuel state by routine or holographic interferometry. The filament lamp (9) illuminated the target during recording its shadowgraph survey. The lens assembly (10) reconstructed the target image in the plane of the film (11). CW laser (12) and shutter (13) were used to evaporate the frozen fuel inside a microsphere. The target arrangement in cryostat is analogous to Ref. 1.

2. The theoretical investigation of the freezing process was based on the numerical solution of a set of equations for thermal conduction, continuity, motion of the phase boundaries, and fuel mass conservation for geometrical arrangement of layers shown Fig. 2. The pressure inside target was assumed identical everywhere and at the condensation onset equal to the saturated vapor pressure. We neglected temperature jumps at phase boundaries except the boundary between the thermal exchange gas and glass. Moreover, we ignored the influence of the interface curvature on the value of saturated vapor pressure. All of the kinetic coefficients were dependent on temperature, and the density of liquid and gas was in addition dependent from pressure. The coefficient values were taken equal to those for DT molecules.

3. The calculated motion of phase boundaries and the experimental points for the width of target outline contour are shown during the freezing process in Fig. 3. The experimental points have been obtained by processing the films of target shadowgraph by microphotometer.

The theoretical distribution of the gas temperature inside a target is presented in Fig. 4. It is shown that the temperature in the target center is smaller than the temperature of saturated vapor after the condensation onset. In this figure also are presented the dependences of the overcooling degree on the target radius and time during the freezing process. In this connection, one can expect nucleation and condensation of the fuel in the microsphere volume, and not on its wall. Fig. 5 shows shadowfilm of targets filled with deuterium and D-T mixture. One can see how the drops form, grow and fall to the microsphere bottom. This is probably a reason of formation of the small structure in the cryogenic layer, showing interferograms of such a cryogenic layer of a different sensitivity obtained by holographic method.²

The cryogenic layer uniformity depends upon the speed of liquid freezing. In Fig. 7a the calculated dependences of the time required for full freezing of the cryogenic layer are presented against the pressure of thermal exchange gas. Here, the uniformity of the cryogenic layer thickness can be estimated from a comparison of the time required for full freezing with the characteristic time during which the liquid layer is flowing down to the target bottom². For example, we have determined the region of r and r values within which the nonuniformity of liquid layer will be smaller than 5%. We have done this using the criterion t/r<20 μm·s. The boundary of this region is shown in Fig. 7b in the coordinate system: the pressure of thermal exchange gas and target ra-
dium \((P_{He-r})\) for the target with the D-T gas pressure of 30 atmosphere and the cryostat temperature of 10 K. It is shown that the nonuniformity of cryogenic layer will exceed always 5\% for the targets with \(r>r_k=250 \mu m\) because of the limited heat transmission by thermal exchange gas.

If one can avoid the growth of large drops on the one hand and liquid flow on the other hand, a uniform layer of solid fuel will be obtained. The interferogram of a target with a cryogenic layer inhomogeneity less than 20\% is shown on Fig. 7c.

4. Since solid uniform layer in big targets with a large amount of fuel cannot be fabricated very rapidly it is inevitable that the fuel will be in the liquid state for a sufficiently large time interval.

The theoretical investigation of conditions, under which it is possible to fabricate a uniform liquid cryogenic layer, has been performed using the diagram and equations given in Fig. 8. These are two-dimensional steady state equations for the temperature of glass, liquid and gas, equations of continuity and motion for liquid and gas. The boundary conditions take into account the full moistening and adhesion of the liquid to glass, flow continuity of the substance, and a change of the thermal flux at the liquid–vapor boundary. The temperature difference inside a target was assumed small, therefore all kinetic coefficients were taken for the average temperature. The equations were solved for two conditions: 1) the thickness of liquid layer was always the same \((r=d)\) and 2) the center of spherical boundary of liquid–vapor was displaced in the vertical direction against the container center \((r=d+\delta \cdot \cos \alpha)\). The coefficient values were taken for deuterium.

The calculated results shown the following. In order to ensure the homogeneity of the liquid layer thickness, it is necessary to create the temperature distribution \(T=T_0+AT-C\cos \phi\) on the glass microsphere surface and the similar distribution of the thermal flow. This can be ensured, when a substance on the outside boundary of the microsphere will have a constant temperature gradient. Such conclusion coincides with that of Ref. 4. The calculated dependence of the desired temperature gradient on the fuel amount inside the target is shown in Fig. 9. The shaded region shows the layer thickness nonuniformity less than 5\%.

The calculated investigation of thermal exchange between a target and cryostat walls has shown that is possible to fulfill exactly the condition required for obtaining a homogeneous layer, when a target is placed in a gas as in continuous medium between two well conducting heat horizontal planes, analogous to Ref. 4. One can approach an ideal case by removing heat through a filament at the top of a target and warming the bottom by molecular flow or thermal radiation, analogous to Ref. 5.

The experimental investigation of the behavior of a liquid D-T mixture layer inside a microsphere has been performed according to a scheme in Fig. 1 with a metal hanging fiber. There have been performed three series of experiments. 1. The target is in "cold" chamber (5). 2. The target is surrounded by shield having the liquid nitrogen temperature (chamber (5) removed). 3. The target is in room temperature environment (chamber (5) and shield (6) are removed). In each series of experiments cold rod (3) in the cryostat is slowly heated (with the rate of 0.2 - 0.3 K/min.). By this a practically steady state is reached for a liquid and vapor. The target is dark and illuminated from time to time by a laser pulse with the duration of ~0.001 s for recording a hologram. After the reconstruction and overlapping of two holograms an interference picture is obtained. The interferograms of liquid layer are shown in Fig. 10.

One can see that in all case the solid fuel is recondensed at the point of fiber contact with the microsphere, that is in the coldest region. In the first series of experiments, as the temperature increases the fuel at first is mainly at the target top and then moves to the bottom. We think the main reason of this is the viscosity decrease with the temperature increase. It is shown also, that a discrepancy between the microsphere pole and the point of fiber contact with it leads to a larger layer nonuniformity over the azimuth angle \(\phi\). At some temperature it is possible to obtain the state when the layer thickness at the top and at the bottom is the same.

In the second case, when the target is surrounded by the shield of the liquid nitrogen temperature, probably it is also possible to create conditions when the liquid fuel layer will be similar to uniform one.
In the third case, when the target is in room environment, the liquid fuel is always collected at the target top. The bottom has the temperature above the boiling temperature and is not moistened.

It should be noted that in the case of a large temperature difference inside a target a spatial separation of isotopes in the mixture is possible, it is not recorded by interferometry.

Theoretical and experimental investigations of thermal exchange between the target and the laser chamber walls in room temperature have shown that a cryogenic layer in the target may not be destroyed during time interval enough for the implosion experiment to be performed. For example, in the target with the radius of 100 μm and fuel amount of 30 amaga the cryogenic layer melts in ~0.1s and evaporates completely in~0.5s after opening the target for power laser beams.

The performed investigations have allowed to design the system and to manufacture its model for the fabrication of the uniform cryogenic layer in the target placed in the working laser chamber. The scheme is shown in Fig.11 and similar to Ref. 7. The gas filled target is placed into the interaction chamber on a holder having good thermal contact with the cold part of cryostat. When the power laser beams are pointed to the target, the latter is covered by a mobile shield which is cooled with helium vapor. Gaseous helium at the pressure up to 1-15 Pa fills the inside of the shield. The fuel freezes nonuniformly. Next the fuel is evaporated by a beam from an auxiliary CW laser by method of fast refreezing proposed by Miller⁶. When this laser beam is stopped, the fuel refreezes uniformly on the inner wall of the microsphere. By this method it is possible to prepare a cryogenic target with a diameter of 100-300 μm and wall thickness of 0.5-2 μm. After formation of a film the mobile shield is moved away rapidly and the target is illuminated by power laser beams before the cryogenic layer will melt.

References

Target arrangement in cryostat

1- glass target filled D-T or D2
2- glass or metal fiber
3- cold rod of cryostat
4- windows for target view
5- chamber with exchange gas(He , H2)
6- shield of the liquid nitrogen temperature
7- walls of room temperature

Equipment arrangement

8- CW- laser for hologram recording
9- filament lamp for shadowgraph recording
10- lens assembly
11- film, holographic plate
12- CW- laser evaporating the frozen fuel
13- shutter
Set of equations for thermal conduction, motion of the phase boundaries, and fuel mass conservation.

\[ c_i \frac{dT_i}{dt} = \frac{1}{x^2} \cdot \frac{d}{dx} \left( x^2 K_i \frac{dT_i}{dx} \right), \quad i = 1, 2, 3, 31, 32 \]

\[ c_{30} \left( \frac{dT_{30}}{dt} + \nu_{30} \frac{dT_{30}}{dx} \right) = \frac{1}{x^2} \cdot \frac{d}{dx} \left( x^2 K_{30} \frac{dT_{30}}{dx} \right) + 0.1013 \frac{dP_{30}}{dt} \]

\[ \text{div} \vec{v}_{30} = 0 \]

\[ q_1 \frac{dX_1}{dt} = K_{31} \frac{dT_{31}}{dx} / x_1, \quad \frac{dT_{30}}{dx} / x_i, \quad T_{30} \leq T_{\text{cond}} \]

\[ q_2 \frac{dX_2}{dt} = K_{32} \frac{dT_{32}}{dx} / x_2, \quad \frac{dT_{31}}{dx} / x_i, \quad T_{31} \leq T_{\text{solid}} \]

\[ \frac{1}{X_1} \int_{0}^{X_1} \rho_{30}(x) \cdot x^2 dx = \frac{r_{in} \rho_{30}}{3} - \int_{0}^{t} \rho_{30}(X_i) \cdot \nu_{30}(X_i) dt \]

1 - room for a thermal exchange gas
2 - glass shell
30 - D-T gas
31 - liquid D-T
32 - solid D-T
4 - thermostat
5 - hanging filament

T - temperature
K - thermal conductivity coefficient
P - pressure
\( \rho \) - density
\( v \) - gas velocity
c - thermal capacity
\( X_1 \) - position of the liquid-vapor boundary
\( X_2 \) - position of the solid-liquid boundary
Fig. 3 RESULTS OF THE CALCULATION OF TARGET FREEZING PROCESS

Calculated motion of the phase boundaries and measured thickness of a cryogenic layer

Temperature of gas inside microsphere.
The temperature in target center is smaller than one of saturated vapor.
Fig. 4  THE RESULTS OF CALCULATION OF TARGET FREEZING PROCESS

\[ \theta = \frac{(T_{\text{sat}} - T)}{T_{\text{sat}}} \quad \theta_0 = \frac{(T_{\text{sat}} - T_0)}{T_{\text{sat}}} \]

\( T_0 \) - temperature on the microsphere center

The overcooling degree on the microsphere center against time

Distribution of the gas overcooling degree inside of microsphere
EXPERIMENTAL INVESTIGATION OF THE TARGET FREEZING PROCESS

The shadowgraphs of targets filled with deuterium and D-T mixture. One can see how the drops form, grow and fall to the microsphere bottom. Filming rate is 48 pictures per second.

D-T mixture
\[ P_{He} = 0.04 \text{ torr} \quad P_{He} = 0.03 \text{ torr} \]

D_2
\[ P_{He} = 0.04 \text{ torr} \quad P_{He} = 0.03 \text{ torr} \]
The interferograms of cryogenic layer of a different sensitivity obtained by holographic method.

The small structure in cryogenic layer is shown.

Probably reason of these is the drops forming.
Fig. 7  THE RESULTS OF INVESTIGATION OF TARGET FREEZING PROCESS

The calculated dependences of the time required for full freezing of the cryogenic layer against the pressure of thermal exchange gas

The calculated boundary of region where the inhomogeneity of liquid layer will be smaller than 5% in the coordinate system - the pressure of thermal exchange gas and target radius

Inhomogeneity of a cryogenic layer less than 20% by avoiding the growth of large drop and liquid flow down
THEORETICAL INVESTIGATION OF CONDITIONS FOR
UNIFORMITY LIQUID CRYOGENIC LAYER

Two-dimensional steady state equations for the temperature of glass, liquid and gas, equations of continuity and motion for liquid and gas.

\[ \Delta \tau_i = 0 \quad i = 1, 2, 3 \]
\[ \text{div} \, \vec{u}_i = 0 \quad i = 2, 3 \]
\[ \nabla^2 \vec{u}_i = P_0^i \cdot \text{grad} \, p_l - G_0^i \quad i = 2, 3 \]

The equations were solved for to conditions:
1) the thickness of liquid layer was always the same
   \[ r = d \]
2) the center of spherical boundary of liquid-vapor was displaced in the vertical direction against the container center
   \[ r = d + \delta \cdot \cos \theta \]

\( \tau \) - dimensionless temperature
\( \vec{u} \) - dimensionless velocity
\( P_0 \) - ratio of pressure and friction force
\( G_0 \) - ratio of gravity and friction force
THEORETICAL INVESTIGATION OF CONDITIONS FOR FORMING A UNIFORMITY LIQUID LAYER

In order to ensure the homogeneity of the liquid layer thickness, it is necessary to create the temperature distribution $T = T + \Delta T \cdot \cos \phi$ on the glass microsphere surface or a constant temperature gradient.

The calculated dependences of the desired temperature gradient on the fuel amount inside of the target.
EXPERIMENTAL INVESTIGATION OF LIQUID LAYER BEHAVIOUR

Holographic interferograms of the liquid layer on the target in different temperature environment.

The target is in cold environment.
Point of the fiber contact is aligned by the target top.

The target is in cold environment.
Point of the fiber contact is replaced from of the target top.
T=19.5K, 20.6K, 22K, 23.7K

The target is surrounded by the shield in liquid nitrogen temperature.
T=11.1K, 21.9K, 24.3K, 26.8K

The target is in room environment
T=22.7K, 23.5K, 24.2K, 25.8K

The heat is conducted through
SYSTEM FOR FABRICATION OF THE UNIFORM CRYOGENIC LAYER ON LASER CHAMBER

1 - target
2 - vacuum
3 - cold rod of the cryostat
4 - cold mobile shield
5 - windows
6 - vapor of the helium
7 - channel for gaseous helium
8 - liquid nitrogen shield
9 - one of power laser beam
10 - beam of an auxiliary laser
11 - view direction
The characteristics of cryogenic ICF capsules containing deuterium-tritium (DT) are greatly influenced by the heat generated by the decay of the tritium. Consequently, it is essential to perform experiments with DT in order to accurately measure characteristics of these capsules. In order to facilitate these measurements we are developing a self-contained DT source to supply gas for these experiments.

The risk of release of DT gas comes primarily through leaks from system components at pressures above atmospheric pressure and from vacuum pump effluent. Our system minimizes these risks by operation below atmospheric pressure at all times, and by the inclusion of a cryogenic sorption pump which produces no effluent. We describe the operation and performance of this system.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Several experiments at NOVA, in the past year, have called for carbon fibers coated with 2000Å of 50/50 Ti/Cr for the use in isoelectronic temperature measurements. These fibers are 5 μm OD and were mounted on a fixture for coating in the PVD rotating coater. We found that the length of the fibers was very critical to the uniformity of the coating. Fibers over 1 cm in length would flex enough during pumpdown to entangle with fixturing. This would destroy the coating geometry and resulted in non-uniform coating as determined by SEM x-rays analysis.

This poster will show how we overcame these difficulties and how the fibers were used in different NOVA targets.
COATING FIBERS

CARBON FIBER
6-9 um OD
10 mm long

COPPER WIRE

TOO LONG
TOROIDAL GAS-FILLED HOHLRAUM

SOUTH VIEW

- stalk
- window frame
- WAX
- thin metal patch
- Cr-Ti coated graphite fibers
- South hole

NORTH VIEW

- North hole

Los Alamos

517
Ti/Cr fiber trapeze

SAMPLE OF A TRAPEZE

JCF LGHOH LONG SLOT
CARBON FIBER PLACEMENT IN BISMUTH REEMIT SPHERES

Joyce Moore, Norman Elliott, Veronica Gomez, and Gerald Rivera

LOS ALAMOS NATIONAL LABORATORY
MATERIALS SCIENCE AND TECHNOLOGY DIVISION
LOS ALAMOS, NEW MEXICO

Bismuth spheres 300μm in diameter are required to be mounted on minimal mass carbon fibers for use in re-emission targets. Two important fabrication requirements are that no joining material of any kind be visible on the outside of the sphere and that the fiber be stiff enough to support the sphere vertically from beneath. A process to produce small blind holes and subsequently join fibers without any exposed glue will be described.
ORIGINAL DESIGN OF REEMISSION TARGET

SPHERE IS SANDWICHED BETWEEN TWO LAYERS OF 800A FORMVAR FILM

EXPERIMENTORS SUSPECTED PERTURBATIONS WERE CAUSED BY TOO MUCH MASS
WE WERE ASKED TO SUSPEND THE SPHERE WITH A CARBON FIBER
Bi SPHERE with 15um wide by 50um deep E.D.M. hole
Bi SPHERE with 15um wide by 50um deep E.D.M. hole

7-9um CARBON FIBER SOLDERED IN HOLE
GLUED IN FIBERS WERE SUCCESSFULLY USED ON FOUR FIELDDED TARGETS

HOWEVER

IT WAS STILL CONSIDERED TO BE TOO MUCH MASS

SO WE TRIED SOLDERS
50%Bi, 25%Pb, 12.5%Sn, 12.5%Cd by weight

Wood's metal
Sputtered Wood's metal

Cerro bend composition
50%Bi, 35%Pb, 15%In by weight

Cerro bend alloy
Sputtered Cerro bend

Status and Future Directions

- Epoxy gluing works but requires care to avoid excess glue

- Improved wetting of solders may be obtained by oxygen plasma ashing of carbon fibers or minimal use of a fluxing agent
Thin polyimide films for laser targets are of current interest as windows for gas filled ICF targets. We needed an in house capability to supply our need for this high strength polymer material. We were made aware that LLNL was getting targets with this film from Lexel Corp. LLNL was developing the capability to make polyimide film to supply some of their needs. This paper will cover recent refinements to the process developed for the production of thin (0.35 to 1 micron) polyimide windows.

We are using Hitachi L100 resin for our films. Early work at LANL centered on producing the polyimide films by spin coating on glass slides coated with VictaWet release agent. While films produced by this method were easily removed, the quality of the films varied. To produce high quality films, silicon wafer substrates were used and the polyamic acid solution was carefully filtered and degassed. The resulting films were cured, mounted and tested for leak tightness, strength, and burst pressure. Details of the process and the results will be presented.
Polyimide Film for Laser Targets

By

Mike A. Salazar, Mike Mitchel, Pete Gobby, Larry Foreman, Rick Behymer*

*Lawrence Livermore National Laboratory
Resin Preparation

• PIQ-L100 resin from Hitachi was thinned with 1 part DMF to 5.7 parts resin solution by weight.
• This solution is then passed through a .2 micron syringe filters to remove large particles. The use of this filter entrains air in the solution.
• We remove the air by centrifuge. This ensures that any entrained air is removed and separates any heavy particles or polymerized globs of material.
• Resin is then removed from the top portion of the mix being careful to introduce no air.

Substrates

Glass slips
• Release agents are attractive to reduce the time it takes to remove film from substate.
• Victawet was vapor deposited.
• It is water soluble.
• Variations in relative humidity cause changes in its surface finish so we quit using it.
Substrates

Silicon Wafers
• Silicon wafers have a very good surface.
• Silicon wafers have high reflectivity.
• Silicon wafers are easily obtained.
• Silicon wafers have a large surface area
• We just happen to have a vacuum chuck just the right size to hold a 2 inch diameter wafer.
• We found that film cut to size could be removed in a reasonable amount of time with hot water and a little mechanical effort.

Spinning
• The wafer is brought up to speed.
• It is flooded it with methanol to wash off any debris that might have fallen onto the wafer.
• The resin is placed onto the stationary surface of the wafer.
• The wafer is brought up to speed rapidly and held at speed for 30 seconds.
• The wafer is removed from the fixture and placed into a clean covered glass dish to keep dust off the surface.
• Cure at 50C for 1 hr., 250C for 1 hr., and 350C for 1 hr.
Spinning

We have made films of various thickness by changing the speed used to spin the wafers. The results of which are represented in the accompanying graph.

Quality Control

- VictaWet release for the Polyimide was subject to variation of its surface smoothness.
- Through visual inspection of films on silicon wafers under magnification we can see areas that have defects.
- Film with defects such as pin holes contain bright spots.
- Film with inclusions has the appearance of rays.
- Continuous film has a uniform dark appearance.
- We selected areas without visible defects for pressure and leak tests.
- These tests have shown that we can successfully select and bond this film in a manner that insures a quality target.
Strength Tests

- We have bonded films of .35 to .93 microns thickness to Gold hohlraum halves.
- We conducted bulge and leak tests at various pressures.
- An optical comparator was used to measure radius of curvature.
- The charts show the results of some of these tests.

Radius of Curvature vs Pressure

Films are mounted flat prior to testing. As pressure is applied the film plastically deforms to progressively smaller radius of curvature.
Pressure vs Thickness

As a film is stretched mass must be conserved. These thickness are calculated assuming the original mass of the film within the LEH is evenly distributed over the stretched, pressurized film and the density remains constant (approximately 1.4g/cm3).

Stress vs Pressure

The first and third data sets were bonded to the edge of the LEH. In the other two data sets films are bonded exterior to the rounded edge of a washer. It seems that the non uniform bonded edge affects negatively the ultimate stress at which films break. A uniform even contact area where film meets metal spreads the load more evenly enhancing the ultimate stress level.
Conclusion

• Polyimide films can be made strong, leak tight, at a variety of thickness.

• Process control is the key to a consistent product.

• Tests show that we can construct targets that will hold considerable pressure.

• Note: Stress at Failure is approximately 2X advertised tensile strength (45ksi). We attribute this to the fact that the film is under bi-axial stress in our tests.
The composition and crystalinity of Si$_x$N$_y$ thin window material used in long scale length plasma experiments was characterized using Analytical Electron Microscopy (AEM). The results are presented and compared to reported values for stoichiometric Si$_3$N$_4$. 
MICROSTRUCTURAL ANALYSIS
OF SILICON NITRIDE WINDOW
MATERIAL

Kerry Siebien
Norman Elliott
Mike Salazar

Los Alamos National Laboratory
MST-7, POLYMERS & COATINGS
Los Alamos, New Mexico
Assembled toroidal hohlraum with fill tube used in long scale length plasma instability experiments.

Gold hohlraum and a silicon nitride window. The window is .3 micron thick with a 50 micron thick silicon frame.

The windows were fabricated by Dino Ciarlo at Lawrence Livermore National Laboratory.
Schematic of the assembled gold hohlraum, silicon nitride window, and fill tube.

SAMPLE PREPARATION

A cross section and plan view of the silicon nitride window were analyzed in the Analytical Electron Microscope. The cross section was prepared by embedding a small piece of the window in epoxy and slicing with an ultra microtome. The plan view was prepared by adhering a window directly to a copper grid.
Microtomed cross section of a silicon nitride window. The window thickness is 300 nm.

Plan view of the silicon nitride window. The material is amorphous, as evidenced by the featureless structure and the diffuse rings in the selected area electron diffraction pattern.
Typical energy dispersive x-ray spectra from the Si3N4 standard.

Typical energy dispersive x-ray spectra from the window material.
ENERGY DISPERSIVE X-RAY RESULTS

<table>
<thead>
<tr>
<th>Analysis Point</th>
<th>Si</th>
<th>N</th>
<th>Si:N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si₃N₄ standard</td>
<td>60.0</td>
<td>40.0</td>
<td>1:1.3</td>
</tr>
<tr>
<td>1</td>
<td>71.3</td>
<td>28.7</td>
<td>1:0.8</td>
</tr>
<tr>
<td>2</td>
<td>68.3</td>
<td>31.7</td>
<td>1:0.9</td>
</tr>
<tr>
<td>3</td>
<td>68.5</td>
<td>31.5</td>
<td>1:0.9</td>
</tr>
<tr>
<td>4</td>
<td>72.5</td>
<td>27.5</td>
<td>1:0.8</td>
</tr>
<tr>
<td>5</td>
<td>73.1</td>
<td>26.9</td>
<td>1:0.8</td>
</tr>
<tr>
<td>6</td>
<td>69.2</td>
<td>30.8</td>
<td>1:0.9</td>
</tr>
<tr>
<td>7</td>
<td>67.8</td>
<td>32.2</td>
<td>1:1.0</td>
</tr>
<tr>
<td>mean</td>
<td>70.1</td>
<td>29.9</td>
<td>1:0.9</td>
</tr>
<tr>
<td>median</td>
<td>69.2</td>
<td>30.8</td>
<td></td>
</tr>
</tbody>
</table>

The Cliff Lorimer equation with absorption corrections and a Si₃N₄ thin film standard were used for the quantification.


CONCLUSIONS

1. Silicon nitride window material is amorphous.

2. The composition of the window material is SiN₀.9, which is non stoichiometric and nitrogen deficient.
MEASURING AND ANALYZING THE PERFORMANCE OF AN ATOMIC FORCE MICROSCOPE-BASED PROFILOMETER*

R.L. McEachern, C.E. Moore, and R.J. Wallace
University of California
Lawrence Livermore National Laboratory
P.O. Box 808, Livermore, CA 94551

Rayleigh-Taylor instabilities during implosions of inertially confined fusion (ICF) capsules affect capsule performance. During acceleration, surface imperfections grow and can, if large enough, lead to an asymmetric implosion or even shell breakup. For this reason, characterizing the topography of target capsules is extremely important. We have developed a profilometer based on an atomic force microscope combined with a precision rotary air bearing. Averaged 1D surface height power spectra obtained with this instrument are converted to 2D mode spectra, which are used as input to LASNEX simulations. Knowledge of the performance limits of the apparatus are essential to gauge the reliability of the data. In addition, the statistical nature of the technique, i.e. characterizing a 2D surface with 1D measurements, must be considered. We will present measurements of the air bearing runout and overall system noise. Using computer simulations of these measurements, we will also discuss the statistics involved in averaging 1D power spectra. Finally, we will show the application of this measurement technique to capsules that have been laser-ablated, resulting in a well-defined surface topography. This special case provides an excellent test for the system, since the expected results are exactly calculable.

* Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.
Introduction

Hydrodynamic instabilities during implosions of inertially confined fusion (ICF) capsules are expected to play a dominant role in determining overall capsule performance.\(^1\) Deviations from sphericity of the as-made capsule surface can provide the seed for such instabilities. In light of the important role played by surface topography in determining hydrodynamic stability, we have developed an atomic force microscope (AFM) based profilometer that gives us the capability to measure equatorial traces on target capsules with nm height resolution. This presentation focuses on the measured performance limits of this instrument and computer simulations of the measurement process.

Performance

Figures 1-3 show photographs of the apparatus, a schematic diagram, and some typical data. Possible sources of random error in these measurements include asynchronous deviations in the motion of the air bearing, vibration of the AFM, thermal drifts, or electrical pick-up. While it is difficult to separate the relative magnitudes of these effects, it is possible to estimate the total contribution from random sources. By repeatedly measuring the same trace on a very smooth capsule, some sense of the reproducibility can be obtained. Figure 4 shows the result of one of these measurements.

The principal source of systematic error is synchronous error motion, or runout, of the air bearing rotor; i.e., wobble motion that repeats each time around. Since a perfect reference sphere does not exist, this error must be deconvoluted from the topography of the measured sample. Briefly, this is accomplished by measuring a trace, or set of traces, on a capsule, rotating it with respect to the vacuum chuck (while maintaining the same polar orientation), and measuring it again. Since the capsule topography will be the same, but shifted in phase, while the synchronous runout will not be phase-shifted, these data allow the runout to be isolated. Figure 5 shows the result of such a measurement.
Measurement Statistics

The characterization of a 2D surface with 1D measurements raises certain issues that are independent of the apparatus. First, the connection between the actual surface topography and the data provided by the measurement must be established. Then, given such a connection, the statistical reliability of the measurement must be determined. The latter goal has been achieved through the use of computer simulations.

The surface topography of a target capsule is typically characterized by a power spectrum \( P_{2D}(l) \), where \( l \) is the mode number associated with a spatial wavelength \( l = \text{circumference/wavelength} \). This is based on a description of the surface height \( Z \) as a sum of spherical harmonics:

\[
Z(\theta, \phi) = \sum_{l=1}^{\infty} \sum_{m=-l}^{l} R_{lm} Y_{lm}(\theta, \phi).
\]

\( P_{2D} \) is then defined as

\[
P_{2D}(l) = \sum_{m=-l}^{l} |R_{lm}|^2.
\]

This power spectrum can be converted to an "average" 1D power spectrum, i.e., the spectrum toward which an average of many 1D spectra (as calculated from individual traces) will converge. The reverse can also be accomplished, which is the basis for deriving 2D power spectra from actual measurements. This raises the issue of determining how well one knows \( P_{1D} \). Since it is difficult to measure enough traces on a real sphere to get a decent picture of the measurement statistics, we use a computer program that simulates the measurement process. Figure 6 shows the results of this simulation using a computer-generated surface. The averaged spectrum clearly converges properly to the calculated one. Furthermore, the histograms shown in Fig. 6b demonstrate that individual measurements of a particular mode can be modeled by an exponential probability distribution. This result can easily be generalized to the typical measurement in which multiple power spectra are averaged together. The general distribution \( P(l_{\text{avg}}) \) for the value of mode \( l \) obtained by averaging \( n \) power spectra is

\[
P(l_{\text{avg}}) = \frac{(an)^n l_{\text{avg}}^{n-1}}{(n-1)!} e^{-anl_{\text{avg}}},
\]

where \( 1/\alpha \) is the mean and \( 1/(\alpha \sqrt{n}) \) is the standard deviation. Note that for \( n=1 \), the standard deviation equals the mean, as demonstrated in Fig. 6.
Application to Ablated Capsules

An ongoing series of experiments is being performed at LLNL that requires the production of target capsules that have a specified 2D power spectrum. This is accomplished by using an excimer laser to ablate pits in the surface of finished capsules. By varying the pattern and depth of these pits, different power spectra can be generated. The computer programs we have written to help determine the characteristics of the sphere mapper have also been applied to the problem of calculating the power spectrum associated with a given pattern of pits. Figure 7 shows two examples of pit patterns and their associated power spectra. These experiments also provide the opportunity to verify the performance of the sphere mapper by providing a surface with a pre-determined topography. Figure 8 shows a comparison between a measurement of an ablated capsule and a simulation that used the same pit profile and pattern.

Conclusion

The profilometer described here has proven to be an effective tool for characterizing the topography of ICF target capsules. We have measured the mechanical performance of the instrument, and by using computer simulations, we have determined the measurement statistics relevant to the process. Finally, we have shown the application of these techniques to laser-ablated capsules, which demonstrates the accuracy of the system.

References

Fig. 2. A schematic view of the sphere mapper apparatus.
Fig. 3. An example of typical data taken from a Ti-doped polystyrene mandrel. Figure 3a shows an equatorial trace of the capsule. Note that the vertical scale is expanded by a factor of almost 2000 compared to the horizontal scale. Figure 3b shows the power spectrum derived from such data. In a typical measurement, three sets of three parallel traces are obtained, with the capsule rotated to an orthogonal orientation for each set. The FFT and power spectrum of each trace is computed, and they are all averaged together to give the curve as shown.
Fig. 4. A power spectrum of the random noise. It is obtained by repeatedly measuring the same trace on a smooth ball, subtracting two successive traces from each other, computing the FFT of the remainder, dividing by \( \sqrt{2} \), and calculating the power spectrum. The curve shown is an average of four such spectra.
Fig. 5. A measurement of the air bearing runout using a precision Si$_3$N$_4$ ball bearing. Ten traces were measured and averaged together, the ball was rotated by 105° with respect to the vacuum chuck, and ten more traces were measured. Figure 5a shows the extracted runout. The high frequency noise is not associated with the bearing. Figure 5b gives the power spectrum derived from averaging several similar measurements. The spectrum is zeroed at mode 7 because that mode is nearly synchronous with 105°, and therefore was not measured.
Fig. 6. (a) The results of calculating power spectra of computer-generated traces. Each trace represents 256 equally-spaced height values around a circumference of a sphere with random surface topography. 300 randomly-oriented traces were generated. The standard deviation values refer to the data from which the average was calculated. The correct 1D spectrum is derived by transforming the 2D spectrum that represents the surface. (b) Histograms showing the distribution of values for three 1D modes, along with exponential curves calculated to give the correct expectation value for each mode. The vertical scaling of the curves is arbitrary.
Fig. 7. Pit patterns and their associated power spectra. Figure 7a shows a pattern of 200 randomly-placed pits, each with a 1-μm-deep, 75-μm-diameter Gaussian profile. The circles represent the approximate pit size. Figure 7b shows a regular array of 92 pits, chosen to give a nearly single-mode surface.
Fig. 8. A comparison between data and calculation for a capsule with a 200 pt random pattern. Figure 8a shows a typical trace measured from a capsule compared with trace generated using a computer simulation of the measurement. Figure 8b shows the measured and calculated 1D power spectra. The measured spectrum is an average of the power spectra of 9 traces.
Surface perturbations, i.e., deviations from perfect sphericity, on ICF capsules result in growth of Rayleigh-Taylor (R-T) instabilities during the acceleration phases of an implosion. To test current theories of Rayleigh-Taylor growth and its effect on target performance, we are investigating methods of applying known perturbations to planar foils and smooth ICF capsules. A successful technique has been excimer laser ablation. We will report on the development of an excimer laser ablation micromachining workstation for ICF capsules and R-T samples. Characterization of excimer laser ablated surfaces of relevant plasma ablators will be described. Ablation patterns that give the desired surface finish are presented. Lastly, we will discuss the characterization and analysis of roughened laser ablated capsules and 3D R-T target foils used in ICF experiments.
IMPOSED PERTUBATIONS OF ICF CAPSULES AND 3D R-T TARGET FOILS USING EXCIMER LASER ABLATION

Target Science & Technology
University of California
Lawrence Livermore National Laboratory

Presented to:
Tenth Target Fabrication Specialists Meeting

Feb. 6-9, 1995
Statement of the Problem

Hydrodynamic instabilities during implosions of inertially confined fusion (ICF) capsules are expected to play a dominant role in determining overall capsule performance. The ablation front is Rayleigh-Taylor (RT) unstable due to hot low-density plasma blow-off pushing against and accelerating the denser, cooler shell. During this acceleration, surface imperfections grow at the ablation front and can, if large enough, lead to an asymmetric implosion or even shell breakup. In light of the critical role these hydrodynamic instabilities play in ICF, significant theoretical and experimental efforts have been focused on understanding them in regimes relevant to implosions. A number of experiments on the Nova laser have measured RT growth of known perturbations on ablatively accelerated foils using both direct and indirect drive. The results of these planar experiments have been adequately simulated using the LASNEX code. While these planar experiments have been quite useful, the ultimate goal is to accurately simulate 3D RT growth in a convergent geometry and determine its effect on capsule performance. In support of these types of experiments we have investigated several methods of applying known perturbations to smooth capsules and 3D periodic patterns to foils.

Techniques to produce surface perturbations in ICF capsules

The anticipated Nova implosion experiments will require modified capsule surface roughness of 0.15 to 1.0 µm RMS, with the bulk of the power in the 10-30 mode range. Several methods to produce perturbations on this scale were initially investigated: modifying process conditions during ablator deposition, micro-sandblasting, polymer mist deposition and embedding defects. Unfortunately, it proved difficult with these techniques to create perturbations with the correct RMS and the relevant "lower mode" surfaces. In addition, some techniques changed other physical characteristics of the capsules.

Because of the limitations of the preceding techniques, we began to explore the use of an excimer laser ablation workstation to controllably pattern capsule surfaces. An important feature of this dry etching process is the lack of detectable thermal damage to the etch substrate. In polymeric systems excited with photons of energy greater than about 3.6 ev(λ < 340 nm), the primary decomposition mechanism is photochemical, i.e., the photons excite chemical bonds to energy levels above the disassociation energy. The breaking of these bonds creates a dense gas of small chain molecules and volatile polymer fragments during the absorption of the laser pulse. The rapid expansion of this gas causes the material to be ejected explosively before transferring heat to the remaining substrate. To minimize thermal damage to the substrate and possible compositional changes in the plasma polymer ablator, the current technique was developed using a 193 nm (ArF) excimer laser. The shorter wavelength was also chosen to give improved ablation depth resolution (etch rate/pulse), since for polymer systems the absorption coefficient increases and the penetration depth decreases at shorter excimer wavelengths.
Excimer Laser Micromachining System

The Figure shows a schematic diagram of the excimer laser micromachining system.

- A Questek 2860 excimer laser produces pulses with a width of 30 ns and a maximum beam energy of 350 mJ at 193 nm (ArF). The energy distribution is relatively flat topped horizontally and gaussian vertically.
- An aperture is used to reduce beam energy and to select a section of uniform intensity from the center of the beam.
- A second aperture (a 200 μm pinhole) forms a mask which is imaged onto the capsule surface by a single fused silica lens.
- A beam splitter diverts 10% of the beam to a UV-sensitive CCD camera, which is part of a laser beam analyzer (LBA).
- A programmable eight-axis positioning system is used to place the capsule surface at the laser focus.
- A computer system controls the laser settings, safety system, and capsule translation.

Gross adjustments in the incident fluence are obtained by inserting optical density filters into the light path immediately before the lens system. Finer adjustments are made by varying the laser output. Pulse to pulse measurements of the beam energy show a standard deviation of 8.0%. This simple projection system produces a 2D Gaussian-like intensity distribution at the sample surface.
Excimer laser ablation behavior and characterization of ICF ablator materials

- The average etch rate per pulse vs. laser fluence was obtained and etch rates were found to obey Beer-Lambert's law. Near threshold, however, the rate varies for the first few pulses due to an incubation effect.

- A surface map of a typical gaussian pit ablated in a plasma polymer film measured using AFM shows a relatively smooth surface with no evidence of melting or the granular textures seen in other laser-ablated polymeric films.

Excimer laser ablation does not modify the remaining ablator material

- SEM's energy dispersive analysis, which probes the stoichiometry in the first few µm of a film, showed no detectable change in the dopant concentration in the ablated region of a plasma polymerized doped coating.

- Radiography measures the correct opacity profile of an ablated sample

(a) Radiograph of an ablated doped plasma polymer coating using a Moly anode at 5 kV.

(b) No compositional changes were seen due to the laser ablation process and calculated pit profile correlated with contact profilometry.
Laser ablation technique allows one to control the final power spectrum of the ICF capsule

- Two patterns of pits, giving both a broad and narrow power spectrum, were used when laser ablating capsules.
- Increasing pit depths increase the RMS roughness for a fixed pattern.

Optical microphotograph of a laser ablated ICF capsule (a) random (broad spectrum), (b) “soccer ball” (narrow spectrum).

Laser ablated capsules characterized by a rotary AFM verify our ability to produce predicted surfaces.

Comparison of experimentally measured 1D power spectrum from AFM equatorial traces with calculated simulations for a laser ablated ICF capsule with 200 random pits.
Excimer laser ablated capsules are used for studying hydrodynamic instability in ICF implosions.

- Surface roughness imposed by precision laser ablation
- Surface RMS 0.015 - 2 μm

3D modes are expected to grow more than 2D modes. We have made 3D single-mode $k = (k_x, k_y)$ foils using laser ablation.

AFM: $\eta_z = 1$ μm

Radiograph: $\eta_z = 2.5$ μm

Radiograph analysis:
- Vertical profile
- Horizontal profile
- Fourier Coef (FD)

We have measured on Nova the 3D Rayleigh-Taylor evolution of such a $k_x=k_y$ single-mode perturbation imposed on a planar CH(Br) foil.

- The perturbation has entered the nonlinear regime for $t>3.6\,\text{ns}$.
Summary

A critical objective of the ICF program is to achieve a better understanding of hydrodynamic instabilities during implosions. The development of a new excimer laser ablation micromachining workstation enables us to accurately and repeatably control the initial roughness of an ICF capsule and produce 3D periodic patterns in foils. This method allows the surface to be modified without affecting the other physical properties of the capsule (ablator density, opacity, etc.). Not only can total roughness be varied but the final power spectrum can be tailored from a random surface to a highly symmetric "single mode" pattern. Laser ablated capsules have been characterized with a newly developed profiling system based on an atomic force microscope verifying our ability to produce predicted surfaces. The power spectra that are derived from these traces may be used as inputs to the LASNEX computer simulation that predicts capsule performance.

\[ \text{Growth = buoyancy - drag:} \]
\[ \rho_1 V \frac{du}{dt} = (\rho_1 - \rho_2)gV - \rho_1 u^2 S \]

[Li, Grove, and Zhang. 1993]

[Takabe, Yamamoto, PRA, 44, 5142 (1991)]

[Hecht, Alon, Shvarts, PF 6, 4019 (1994)]
The fabrication of the plastic cylinders used in recent cylindrical implosion experiments at NOVA are presented.

Nine steps were required (five machining, three coating, and one leaching) for the successful completion of a single cylinder. The coating steps included polystyrene, chlorinated polystyrene, brominated polystyrene, and parylene N. Precision machining was required to resolve a marker layer and 30 facets around the outside diameter. The facets were cut around a 520 µm diameter, 1/3 of the facets are 1.83 µm deep. The remaining 2/3 are cut 1 µm deep.
PLASTIC CYLINDER IMPLOSION EXPERIMENT

Gerald Rivera
Leander J. Salzer
Peter L. Gobby
Veronica M. Gomez
Norman E. Elliott

Los Alamos National Laboratory
MST-7, POLYMERS & COATINGS
Los Alamos, New Mexico
WHAT WERE WE ATTEMPTING TO DO?

1. FIVE MACHINING OPERATIONS
2. FOUR COATING OPERATIONS
3. FACETS - INSTABILITIES
4. CHLORINE MARKER - ABSORBS X-RAYS MORE READILY

Los Alamos

Mandrel for diamond turning the perturbed cylinder

Spring loaded spindle adapter mounted on Pneumo lathe spindle.

Diamond turned indicating and touch-off surfaces used for aligning mandrel during each set-up.

1/8" Aluminum stock inserted into "mandrel alignment adapter" and rough turned to within .006" inch on a conventional lathe prior to 1st diamond turning operation.
Part compared to a .7mm dia. lead pencil

View A

10x View A
CONCLUSION

1. MULTI-MACHINING OPERATIONS:
   We were able to do five machining operations, the part was removed from the fixture and re-indicated three times within .000010/inch.

2. FACETS:
   Incorporate a flycutter at 90° from spindle, an indexing ring was designed and fabricated to index 30 positions or every 12°.

3. DIFFERENT COATINGS:
   (1st) Doubly chlorinated polystyrene
   (2nd) Parylene N. water insoluble plastic, to protect the marker during 3rd coating of br-polystyrene, 4th coating of polystyrene.
   Prevent dissolution (blending) of the marker layer.

Los Alamos
520um DIAMETER, 12 FACETS
MACHINING SUB-MILLIMETER BERYLLIUM AND ALUMINUM PARTS FOR THE SPALL TARGETS

Leander J. Salzer, Veronica M. Gomez, Joyce E. Moore,
Jacob J. Bartos, and Peter L. Gobby
Los Alamos National Laboratory
P.O. Box 1663 MS E549, Los Alamos, NM 87544

This poster will show the development of tooling, miniature boring tools, machining steps, etc., required in the machining operations for the sub-millimeter beryllium and aluminum components of the Spall Targets. This target was built for experiments at the HELEN laser at AWE.
MACHINING SUB-MILLIMETER BERYLLIUM AND ALUMINUM PARTS FOR THE SPALL TARGETS

Leander J. Salzer
Jacob J. Bartos
Veronica M. Gomez
Joyce E. Moore
Peter L. Gobby

Los Alamos National Laboratory
MST-7, POLYMERS & COATINGS
Los Alamos, New Mexico
MACHINING BERYLLIUM CAN

The aluminum / beryllium interface required a maximum clearance of 1 micron at the 400 um. diameter plug area. Gaps elsewhere also were not allowed, therefore the concentricity, wall thickness and surface finish tolerances were important.

The machine used for the finish turning operation was our Pneumo Ultra 2000 air spindle machine.

Material used was beryllium stock roughed to 1/16" dia., pre-drilled and c/bored undersize.

The task of boring 800 um. and 400 um. dia.'s and facing the c/bore required designing and fabricating a special tool.

After the boring, turning and parting operations a secondary facing operation was required.

MACHINING ALUMINUM INSERT

The finish turning on the aluminum insert also was done on our Pneumo Ultra 2000 diamond turning machine. 6061 aluminum stock 1/16" dia. was diamond turned to finish dimensions and parted off.

A secondary facing operation was required to complete the part.

On the secondary facing operation, 3 aluminum parts are fastened to a parallel mounting fixture by melting wax around them. This assembly is held in place on a fixture mounted on the lathe spindle and faced to length.
TOOL SETUP FOR BERYLLIUM CAN ON PRECISION LATHE

In order not to lose parts during parting operation, part was not completely separated on machine. After removing stem from collet the part is gently separated with a surgical blade.

SPECIAL BORING TOOL DESIGN

The tool was fabricated using recycled 1/8" dia. solid carbide end mills. To keep the tool (cutting edge) as strong as possible the clearance angle was ground at 24 degrees included angle.

To allow for easy top lapping, the top of the tool was set at 30 degree angle from shank C/L. These angles are built into the grinding fixture.
The boring tool grinding fixture is designed in such a way that all grinding and top lapping operations were performed without removing tool from fixture.

The shank diameter is inserted into the grinder collet.

This surface is used as the base for top lapping.
ICF activities on beryllium incorporate developmental investigations on forming Be hollow spheres. The hollow spheres should have an approximate diameter of 2 mm and a wall thickness of ~100 μm, with an inner and outer surface roughness on the order of 1 μm. In addition, higher atomic number elements are to be alloyed with the Be (~1 at.%) to increase the opacity of the shells. An appropriate alloying element is copper since Cu is soluble in Be and uniform microstructures are required across the wall thickness. The shells will be filled gas, and fabrication efforts should account for the filling process.

Two methodologies for fabricating the Be hollow spheres are being explored: physical vapor deposition (PVD) and drop tube processing. The primary emphasis is on PVD. Two tool steel mandrel have been machined with a hemispherical indent (~2 mm diameter) in the middle of each mandrel. The mandrels were a matched pair that could be aligned to fit together with precision pins so that the two hemispheres would couple together to form a sphere. A Be-Cu alloy was vapor deposited onto both mandrels, which had been masked with aluminum sheet to allow deposition only in a diameter around the indents. During PVD, a small amount of nitrogen was leaked into the system at regular intervals to disrupt the epitaxial growth during deposition. The future intent of this process is to diffusion bond the deposited hemispheres. The bonding can be accomplished within a high gas pressure environment to fill the spheres with gas, eliminating drilling and plugging operations typically associated with gas filling.

In addition to the diffusion bonding of PVD hemispheres, evaluations on the feasibility of drop tube processing have initiated with fluid flow modeling. Using concentric tube nozzle designs, the range of hollow sphere formation is being diagnosed. The drop tube technique offers the capability of filling spheres with gas in an in-situ fashion. For example, the pressure in the drop tube can be adjusted to correspond to the desired fill pressure in the hollow sphere.
Objective

The goal of the ICF activities on beryllium incorporates developmental investigations on forming Be hollow spheres. The hollow spheres should have an approximate diameter of 2 mm and a wall thickness of ~100μm, with an inner and outer surface roughness on the order of 1μm. In addition, higher atomic number elements are to be alloyed with the Be (~1 at.%) to increase the opacity of the shells. An appropriate alloying element is copper since Cu is soluble in Be. Uniform microstructures are required across the wall thickness. The shells will be filled with 400 to 500 atm of gas, and fabrication efforts should account for the filling process.

Methodology

Two methodologies for fabricating the Be hollow spheres are being explored: physical vapor deposition (PVD) and drop tube processing. The primary emphasis is on PVD. For the PVD process, Be-0.9at.%Cu alloys (56 grams) have been arc-cast and machined into sputter targets. The size of these targets are 2 inches in diameter and 3/16 inch thick. Two tool steel mandrels were machined with a hemispherical indent (~ 2mm diameter) in the middle of each mandrel (Figure 1). The mandrels were a matched pair that could be aligned to fit together with precision pins so that the two hemispheres would couple together to form a sphere. In this fashion, deposited hemispheres can be diffusion bonded together. Moreover, the bonding can be accomplished within a high gas pressure environment to fill the spheres with gas, eliminating drilling and plugging operations typically associated with gas filling.

The Be-Cu alloy was vapor deposited onto both mandrels, which had been masked with aluminum sheet to allow deposition only in a diameter around the indents. During PVD, a small amount of nitrogen was leaked into the system at regular intervals to disrupt the epitaxial growth during deposition. The results of the first deposition will be described in the results section.

Evaluations on the feasibility of drop tube processing have initiated with fluid flow modelling. Using concentric tube nozzle designs developed at the Jet Propulsion Laboratory, the range of hollow sphere formation is being diagnosed. The drop tube technique offers the capability of filling spheres with gas in an in-situ fashion. For example, the pressure in the drop tube can be adjusted to correspond the desired fill pressure in the hollow sphere. Preliminary results on fluid flow modelling will be addressed in a later update.

Results

Diffusion Bonding

Diffusion bonding of two identical alloys eliminates the chemical potential driving force typically associated with diffusion. However, random diffusion still exists with thermal activation. Samples of Be-0.9at.%Cu were physically clamped together, encapsulated in vacuum, and soaked at 750°C for three and six hours. Micrographs of the bonds are shown in Figure 2. Sample preparation proved to be vital in establishing a good bond. For example, if the surface finish did not include a final polish on the order of 6 μm or less, bonding would not occur. Also, if the surface flatness was not uniform (e.g. rounded edges), point contacts would occur. As a result, the pieces would bond together at points, degrading the strength of the bond.
Examples of unsuccessful diffusion bonds suggest insight into mechanisms to optimize the microstructures across a bond. For example, Figure 3 shows two micrographs of the Be-Cu samples in a bonding experiment. A good bond was not established, but the microstructure shows signs of recrystallization. The recrystallization could have resulted from a variety of mechanisms, the most plausible of which would be residual stresses at the interface. Recrystallization across an interface during bonding would yield optimal microstructures. Further studies are in progress to evaluate this phenomenon, particularly since sputter deposited samples have residual stresses.

**Sputter Deposition**

The Be-Cu alloy was sputter deposited onto the tool steel mandrel as well as the aluminum mask. A sodium chloride wash was placed on the mandrel to facilitate removal of the deposit. After deposition, warping and cracking was observed in the sample resulting from the residual stresses in the sample. In fact, the integrity of the deposit around the indent lip was questionable on both mandrels. On both mandrels, the deposit lifted off of the tool steel at the lip, leaving the hemisphere inside the impression. A sharp edge in a deposited sample can cause migration of metal away from the corner, reducing the uniformity of the deposit at the lip and permitting decreased structural integrity. An SEM micrograph of a corner of both mandrels is shown in Figure 4. The edge is very rough, and the growth direction is normal to the mandrel, supporting the migration of metal away from the corner.

The Be-Cu alloy was sputter deposited onto both aluminum and the sodium chloride coated tool steel. Cross-sectional views of the sputter deposits on Al are shown in Figure 5. The deposits show the growth of the microstructure normal to the substrate. The structure is disrupted with the nitrogen exposure layers. The level of epitaxial disruption will be determined later with electron microscopy. The micrographs also illustrate that segregation is occurring, and the copper is not uniformly distributed within the deposit. Back-scattered electron microscopy should delineate the more clearly.

Similar microstructures for the deposits onto the tool steel are illustrated in Figure 6. However, one clear distinction exists in that the last nitriding layer has cracked in many locations. The cracked layer suggests one possible mechanism for the brittle tendencies of the deposits.

Since the surface roughness is an important criteria, SEM studies of the surface have been initiated (Figure 7). A surface photograph of the deposit in the bottom of the hemispherical indents displays the surface asperities as well as an observable crack. In addition, “dirt” on the surface is apparent. Although the samples were always stored in bags (except for transfer to the SEM) significant dirt is apparent by the charging species on the sample.

**Discussion**

The sputter deposition process onto a “female” mandrel (i.e., an indented hemisphere) had three main flaws. First, the lips at the corners of the indents did not have any structural integrity, most likely due to migration of the metal away from the sharp edge. As a result, the edges of the hemisphere are rough. Second, the residual stresses were very high in the sample, causing warping and cracking. Third, the nitrogen leaks broke up the epitaxial growth, but also provided an easy crack growth site. Other more minor problems existed, but should be studied.
further to be remedied. For example, segregation is apparent in the sample, but this will be eliminated during a bonding sequence. If the residual stress can be reduced to eliminate cracking, any remaining residual stress will assist in the recrystallization to a more uniform structure of a bonded sphere. Grain growth should be evaluated to eliminate grain boundaries across the wall thickness. Cleaner environments will reduce the collection of dust or dirt on the surfaces. With clean and smooth surfaces, adequate bonds should be possible with three or six hour heat-treatments at 750°C.

Recommendations

The current results suggests the following course of studies which are currently underway:
1. More bonding studies for a proficient evaluation of the bonded microstructures.
2. Both SEM and TEM studies of the sputter deposits to analyze more fully the microstructural development (i.e., crystal structure, interface behavior, segregation patterns)
3. Machine “male” mandrels as opposed to female mandrels to eliminate sharp edge migration.
4. Grain growth and recrystallization studies.
5. Heat-treat sputter deposits to evaluate microstructural development.
Figure 1 - Macrograph of tool steel mandrels with hemispherical indents

Figure 2 - Diffusion bonded Be-Cu at 750°C for a) 3 hours and b) 6 hours.
Figure 3 - Unsuccessful diffusion bonds of Be-Cu at 750°C for a) 3 hours and b) 6 hours.

Figure 4 - Sputter deposits at the corner of the hemispherical indent.
Figure 5 - Micrographs of PVD deposits on the aluminium mask, where a) unetched and b) etched.

Figure 6 - Micrographs of PVD deposits on the tool steel mandrel with a) unetched and b) etched.
Figure 7 - a) Low magnification and b) high magnification micrographs of the surface of the PVD deposits at the bottom of the hemispherical indents.
AGENDA
Tenth Target Fabrication Specialists' Meeting
Taos, New Mexico

February 6-9, 1995

Monday, February 6, 1995

8:15  Welcome  L. Foreman  LANL
8:20  Overview of US ICF Program  G. York  DOE/ICF
8:50  Technologies for Ignition: the NIF  M. Campbell  LLNL
9:20  Omega Upgrade target requirements  H. Kim  UR/LLE
9:40  Overview of Nike laser at NRL  J. Sethian  NRL
10:00  BREAK

Shells I
SESSION Chair:  H. KIM  UR/LLE

10:10  Composite shell production at GA  W. Miller  GA
10:30  Production of large shells by controlled mass encapsulation  L. Cheung  STI
10:50  Prediction of phase separation during drying  G. Wilemski  LLNL
11:10  Use of a ballistic furnace for production of large shells  Nikitenko  LPI
11:30  Fabrication of shells using a depolymerizable mandrel  S. Letts  LLNL
11:50  PHOTO / LUNCH

Shells II
Session Chair:  D. Schroen-Carey  WJSA

1:30  Progress in development of foam shells  G. Overturf  LLNL
1:50  Gas levitation for non-contact coating of balls  K. Kim  U of IL
2:10  Feasibility of organo-beryllium target mandrels  R. Brusasco  LLNL
2:30  Destructive tester for fill and composition  R. Mangano  GA
2:50  BREAK
**Characterization I**

**Session Chair:**

<table>
<thead>
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<tr>
<td>3:00</td>
<td>Design and operation of sphere mapper</td>
<td>C. Moore</td>
<td>LLNL</td>
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<tr>
<td>3:20</td>
<td>Wall thickness using self-interference fringes</td>
<td>M. Wittman</td>
<td>UR/LLE</td>
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<tr>
<td>3:40</td>
<td>Light scattering limitations in characterization of foam shells</td>
<td>R. Stephens</td>
<td>GA</td>
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<td>4:00</td>
<td>The photon tunneling microscope as a characterization tool</td>
<td>C. Hendricks</td>
<td>WJSA</td>
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**Characterization II**

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<td>4:30</td>
<td>Characterization of plates and foam in crack experiment</td>
<td>N. Elliott</td>
<td>LANL</td>
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<td>4:50</td>
<td>Hydrogen out-gassing rates using x-ray fluorescence</td>
<td>D. Steinman</td>
<td>GA</td>
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<td>5:10</td>
<td>X-ray fluorescence calibration</td>
<td>M. Hoppe</td>
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<td>Emissivity and accommodation coefficient of polymer target shells...</td>
<td>A. Honig</td>
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**Tuesday, February 7, 1995**

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<td>8:30</td>
<td>Conference Business</td>
<td>Foreman</td>
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<td>8:40</td>
<td>Stability criteria for high gain polymer capsules</td>
<td>S. Pollaine</td>
<td>LLNL</td>
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<td>9:10</td>
<td>Indirectly driven targets for the National Ignition Facility</td>
<td>D. Wilson</td>
<td>LANL</td>
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<td>9:40</td>
<td>Discussion</td>
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<td>9:50</td>
<td><strong>Break</strong></td>
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**Cryo I**

**Session Chair:**

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<td><strong>BREAK</strong></td>
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10:00 Preliminary experiments in a toroidal cell  J. Hoffer  LANL
10:20 Point spread function measurement  J. Simpson  GA
10:40 Progress toward cryotarget formation  E. Koreshova  LPI
11:00 Structure of vapor deposited solid hydrogen  R. Collins  LLNL
11:20 Results of thermal gradient layering  D. Bittner  WJSA
11:40 LUNCH

Cryo II
Session Chair  J. Sanchez  LLNL
1:10 Theoretical insight of thermal gradient layering  M. Monsler  WJSA
1:30 Resealable cryogenic seals  N. Alexander  GA
1:50 Resonant cavity for plasma heating experiments  H. Kim  UR/LLE
Poster Session I Co-Chairs  K. Shillito WJSA
        M. Wittman  UR/LLE
2:10 Poster Session I Previews
2:30 BREAK/Poster Session I
4:45 ADJOURN
6:30 RECEPTION--BANQUET

Wednesday, February 8, 1995

8:00 Conference Business
International Fabrication
Session Chair  J. Sethian  NRL
8:10 Microtarget fabrication  A. Coudeville  CEL-V
8:40 ICF target technology in the Russian Federal Nuclear Center  V. Izgorodin  VNIEF
4:16 ARZ-16
9:10 Production and characterization of EOS targets  T. Tyrrell  AWE
9:40 Poster Preview II
Session Co-Chairs  J. Moore  LANL
        G. Stone  LLNL

595
Midday Break

6:00 POSTER Session II

8:30 ADJOURN

Thursday, February 9, 1995

8:15 Conference Business

Fabrication I
Session Chair I. Schneir GA

8:20 Low density doped foams M. Mitchell LANL

8:40 Strength and other information on polyimide films E. Hsieh LLNL

9:00 Ion-beam machining B. Lamartine LANL

9:20 Effect of micromachining variables on copper surface finish J. Kaae GA

9:40 BREAK

Fabrication II
Session Chair M. Spragge LLNL

10:00 Current developments / problems P. Gobby LANL

10:20 Miniature targets for high energy density H. Lewis LLNL

10:40 Gas filled hohlraums M. Salazar LANL

11:00 Fabrication and testing of gas-filled hohlraums G. Stone LLNL

11:20 LUNCH

12:45 Workshops
Shells W. Miller GA
Fabrication P. Gobby LANL
Cryogenics J. Hoffer LANL

2:30 ADJOURN
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<tr>
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<th>Organization</th>
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<th>Phone</th>
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<tbody>
<tr>
<td>Thomas Alberts</td>
<td>W.J. Schafer Associates, Inc.</td>
<td>303 Lindbergh Ave.</td>
<td>Livermore, CA 94550</td>
<td>510-447-0555</td>
</tr>
<tr>
<td>Neil Alexander</td>
<td>General Atomics</td>
<td>P.O. Box 85608</td>
<td>San Diego, CA 92186-9784</td>
<td>619-455-2019</td>
</tr>
<tr>
<td>Wallace Anderson</td>
<td>Los Alamos National Laboratory</td>
<td>P.O. Box 1663, MST-7, MS E549</td>
<td>Los Alamos, NM 87545</td>
<td>505-665-3981</td>
</tr>
<tr>
<td>Janet Ankney</td>
<td>General Atomics</td>
<td>P.O. Box 85608</td>
<td>San Diego, CA 92186-9784</td>
<td>619-455-2968</td>
</tr>
<tr>
<td>Elfino Armijo</td>
<td>Los Alamos National Laboratory</td>
<td>P.O. Box 1663, MST-7, MS E549</td>
<td>Los Alamos, NM 87545</td>
<td>505-667-9665</td>
</tr>
<tr>
<td>Mary Lou Atencio</td>
<td>Los Alamos National Laboratory</td>
<td>P.O. Box 1663, MST-7, MS E549</td>
<td>Los Alamos, NM 87545</td>
<td>505-665-1337</td>
</tr>
<tr>
<td>Jacob Bartos</td>
<td>Los Alamos National Laboratory</td>
<td>P.O. Box 1663, MST-7, MS E549</td>
<td>Los Alamos, NM 87545</td>
<td>505-667-9666</td>
</tr>
<tr>
<td>Welsey Baugh</td>
<td>General Atomics</td>
<td>P.O. Box 85608</td>
<td>San Diego, CA 92186-9784</td>
<td>619-455-2096</td>
</tr>
<tr>
<td>Ricke Behymer</td>
<td>Lawrence Livermore National Laboratory</td>
<td>P.O. Box 808, L-474</td>
<td>Livermore, CA 94550</td>
<td>510-423-2650</td>
</tr>
<tr>
<td>Donald Beighley</td>
<td>General Atomics</td>
<td>P.O. Box 85608</td>
<td>San Diego, CA 92186-9784</td>
<td>619-455-2562</td>
</tr>
<tr>
<td>Brian Benicewicz</td>
<td>Los Alamos National Laboratory</td>
<td>P.O. Box 1663, MST-7, MS E549</td>
<td>Los Alamos, NM 87545</td>
<td>505-665-0101</td>
</tr>
<tr>
<td>Thomas Bernat</td>
<td>Lawrence Livermore National Laboratory</td>
<td>P.O. Box 808</td>
<td>Livermore, CA 94551</td>
<td>510-422-5915</td>
</tr>
<tr>
<td>Donald Bittner</td>
<td>W.J. Schafer Associates, Inc.</td>
<td>303 Lindbergh Ave.</td>
<td>Livermore, CA 94550</td>
<td>510-447-0555</td>
</tr>
<tr>
<td>Karl Boline</td>
<td>General Atomics</td>
<td>P.O. Box 85608</td>
<td>San Diego, CA 92186-9784</td>
<td>619-455-4548</td>
</tr>
<tr>
<td>Nataliya Borisenko</td>
<td>Lebedev Physical Institute</td>
<td>53 Lerinskiy Prospect</td>
<td>Moscow 117333</td>
<td>7 095 1326121</td>
</tr>
</tbody>
</table>
Paul Brooks
Los Alamos National Laboratory
P.O. Box 1663
MST-7, MS E549
Los Alamos, NM 87545
505-667-9655

Lloyd Brown
General Atomics
P.O. Box 85608
San Diego, CA 92186-9784
619-455-3078

Raymond Brusasco
Lawrence Livermore National Laboratory
P.O. Box 808
Livermore, CA 94551
510-422-3111

Steve Buckley
Lawrence Livermore National Laboratory
P.O. Box 808, L-474
Livermore, CA 94550
510-422-0938

John Burmann
W.J. Schafer Associates, Inc.
303 Lindbergh Ave.
Livermore, CA 94550
510-447-0555

Harry Bush
Los Alamos National Laboratory
P.O. Box 1663
MST-7, MS E549
Los Alamos, NM 87545
505-667-1986

Helen Bustos
Los Alamos National Laboratory
P.O. Box 1663
MST-7, MS E549
Los Alamos, NM 87545
505-667-7541

Frank Carey
W.J. Schafer Associates, Inc.
303 Lindbergh Ave.
Livermore, CA 94550
510-447-0555

Brendan Casey
W.J. Schafer Associates, Inc.
303 Lindbergh Ave.
Livermore, CA 94550
510-447-0555

Gordon Chandler
Sandia National Laboratories
P.O. Box 5800, MS 1196
Albuquerque, NM 87185-1196
505-845-8384

Gilbert Collins
Lawrence Livermore National Laboratory
P.O. Box 808, L-482
Livermore, CA 94551
510-423-2204

Robert Cook
Lawrence Livermore National Laboratory
P.O. Box 808
Livermore, CA 94551
510-422-3117

Robert Day
Los Alamos National Laboratory
P.O. Box 1663, MS E549
Los Alamos, NM 87545
505-667-2957

Anthony Demiritis
Lawrence Livermore National Laboratory
P.O. Box 808, L-330
Livermore, CA 94551
510-424-6261

Steve Dropinski
W.J. Schafer Associates, Inc.
303 Lindbergh Ave.
Livermore, CA 94550
510-447-0555

Raymond Dukart
Sandia National Laboratories
P.O. Box 5800
Dept. 1271, MS 1187
Albuquerque, NM 87185
505-845-7276

Christian Duriez
CEA, CEL-V
94195 Villeneuve Saint-Georges
Cedex FRANCE
45956963
Bernard Hircq
Commissariat a L'Energie Atomique
DETN - Bat GM - BP No 12
91680 Bruyeres-le-Chatel
France
1 6926 4010

Jim Hoffer
Los Alamos National Laboratory
P.O. Box 1663
MST-10, MS K764
Los Alamos, NM 87545
505-667-4049

Eric Honea
Lawrence Livermore National Laboratory
P.O. Box 808
Livermore, CA 94551
510-422-8118

Arnold Honig
Syracuse University
Physics Department
Syracuse, NY 13244
315-443-3888

Martin Hoppe
General Atomics
P.O. Box 85608
San Diego, CA 92186-9784
619-455-2793

Colin Horsefield

Edmund Hsieh
Lawrence Livermore National Laboratory
P.O. Box 5508, L-482
Livermore, CA 94550
510-422-0753

David Husband
General Atomics
P.O. Box 85608
San Diego, CA 92186-9784
619-455-2971

Vladimir Izgorodin

Roy Johnson
Lawrence Livermore National Laboratory
P.O. Box 808
Livermore, CA 94551
510-422-7254

James Kaae
General Atomics
P.O. Box 85608
San Diego, CA 92186-9784
619-455-2957

Hyo-gun Kim
University of Rochester
Laboratory for Laser Energetics
250 East River Road
Rochester, NY 14623
716-275-5850

Kevin Kim
Univ. of Illinois at Urbana-Champaign
Dept. of Electrical & Comp. Eng.
1406 West Green Street
Urbana, IL 61801
217-333-7162

Elena Koresheva
Lebedev Institute
Russian Academy of Sciences
Moscow, Russia

Bruce Lamartine
Los Alamos National Laboratory
P.O. Box 1663
MST-7, MS E549
Los Alamos, NM 87545
505-665-2366

Stephen Lambert
Soane Technologies, Inc.
3916 Trust Way
Hayward, CA 94545
510-293-1850

Ramon Leeper
Sandia National Laboratories
P.O. Box 5800, MS 1196
Albuquerque, NM 87185-1196
505-845-7185

Stephan Letts
Lawrence Livermore National Laboratory
P.O. Box 808, L-482
Livermore, CA 94550
510-422-4373
Jean Stark
Los Alamos National Laboratory
P. O. Box 1663
PA-4, MS P366
Los Alamos, NM 87545
505-665-2817

David Steinman
General Atomics
P.O. Box 85608
San Diego, CA 92186-9784
619-455-2879

Richard Stephens
General Atomics
P.O. Box 85608
San Diego, CA 92186-9784
619-455-3863

Gary Stone
Lawrence Livermore National Laboratory
P.O. Box 808
Livermore, CA 94551
510-423-0987

Masaru Takagi
ILE Osaka University
2-6-Yamada-Oka
Osaka 565 JAPAN
06-879-8780

S. M. Tolokonnikov
Lebedev Institute
Russian Academy of Sciences
Moscow, Russia

Robert Turner
Lawrence Livermore National Laboratory
P.O. Box 808
Livermore, CA 94551
510-422-8112

Tony Tyrrell
Aldermaston Weapons Establishment
Aldermaston Reading, RG7 4PN
United Kingdom

Russell Wallace
Lawrence Livermore National Laboratory
P.O. Box 808
Livermore, CA 94551
510-423-7864

Wane Wier
General Atomics
P.O. Box 85608
San Diego, CA 92186-9784
619-455-2614

Gerald Wilemski
Lawrence Livermore National Laboratory
P.O. Box 808, L-482
Livermore, CA 94551-9900
510-422-7919

Douglas Wilson
Los Alamos National Laboratory
P.O. Box 1663
XTA, MS B220
Los Alamos, NM 87545
505-667-6154

Mark Wittman
University of Rochester
Laboratory for Laser Energetics
250 East River Road
Rochester, NY 14623
716-275-9524

George York
Los Alamos National Laboratory
P.O. Box 1663
DX-13, MS P940
Los Alamos, NM 87545
505-667-2553