LA-UR-19-30137
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Title: Compilation of DOE Manual 441.1-1 Working Group Meeting Presentations

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Smith, Paul Herrick

Intended for: Documentation of Working Group Meeting
Report

Issued: 2019-10-10 (rev.1)

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Compilation of DOE Manual 441.1-1 Working Group Meeting Presentations

Los Alamos National Laboratory, Los Alamos, NM
August 20-21, 2019

This is a compilation of the presentations at the DOE Manual 441.1-1 Working Group Meeting held at Los Alamos National Laboratory, Los Alamos, NM on August 20-21, 2019. The objective of the meeting was to focus on the complex-wide implementation of DOE M 441.1-1, to include nuclear material storage containers (LANL as well as Y-12 developed) approval for use, procurement, quality assurance, and surveillance. The Technical Basis Document review, lifetime extension of limited life components, certification extension of the SAVY-4000 series of containers was also covered as well as final edits to the Nuclear Material Packaging Standard.

DOE Manual 441.1-1 Working Group Meeting Instructions and Agenda

Los Alamos National Laboratory, Los Alamos, NM
August 20-21, 2019

Objective: Focus on the Complex-wide implementation of DOE M 441.1-1, to include nuclear material storage containers (LANL as well as Y-12 developed) approval for use, procurement, quality assurance, and surveillance. Focus will also include the Technical Basis Document review and lifetime extension of limited life components. Certification extension of the SAVY-4000 series of containers will also be covered as well as final edits to the Nuclear Material Packaging Standard currently being developed.

Location: LANL TA-35, Building 86, Room 205 (HSPD-12 is required to get to TA-35)

- **Pecos Rd off of Pajarito Rd—Google Maps screenshot below with a star on bldg. 86 and the parking lot marked (if that parking lot is full, just drive further down Pecos to find an open spot)**
- **If you search "Pecos, Los Alamos, NM" in Google Maps, the location provided will be a point just down the road from the meeting location**

Badging for PF-4/Classified tour: If you will be on the PF-4 tour on Wednesday, ensure you stop at the Los Alamos badge office to get a PIN. Those participating in the unclassified tour do not need to do this.

WebEx: Those people calling in/attending remotely should be able to view presentations via WebEx

POCs: Paul Smith (505) 699-7554, James Shaw (443) 254-4470

Agenda:

Tuesday, August 20th

07:00 am: Badge office opens (search Google Maps for "Los Alamos Badge Office")

08:30 am: Welcome and Introductions – James Shaw/Paul Smith

LANL Presentations (morning session)

08:40 am: SAVY Update and Design Life Extension – Paul Smith/Tim Stone

09:00 am: SAVY/Hagan Corrosion Update – Josh Narlesky

09:20 am: SAVY/Hagan Accelerated Aging – Daniel Rios/Juan Duque

09:40 am: SAVY Inventory Planning and Procurement – Zachary Smith

10:00 am: Capped SAVY Pressurization Testing – David Grow

Break

10:30 am: PVC Replacement – Joseph Dumont

10:50 am: Modular Integrated Non-Destructive Test Setup (MINTS) – Raj Vaidya

11:10 am: Use of Robotics in Future Surveillance – David Grow

11:30 am: Glovebox Container Development and Testing – Jude Oka

11:50 am: Respirable Release Fraction Measurement Chamber (RRFMC) – Yong Tao/Murray Moore

Lunch

01:30 pm: Site Updates (15 min each)

03:00 pm: NMP Standard Writing Team Meeting and Open Discussion – James Shaw

5:00 pm Adjourn

Wednesday, August 21, 2019

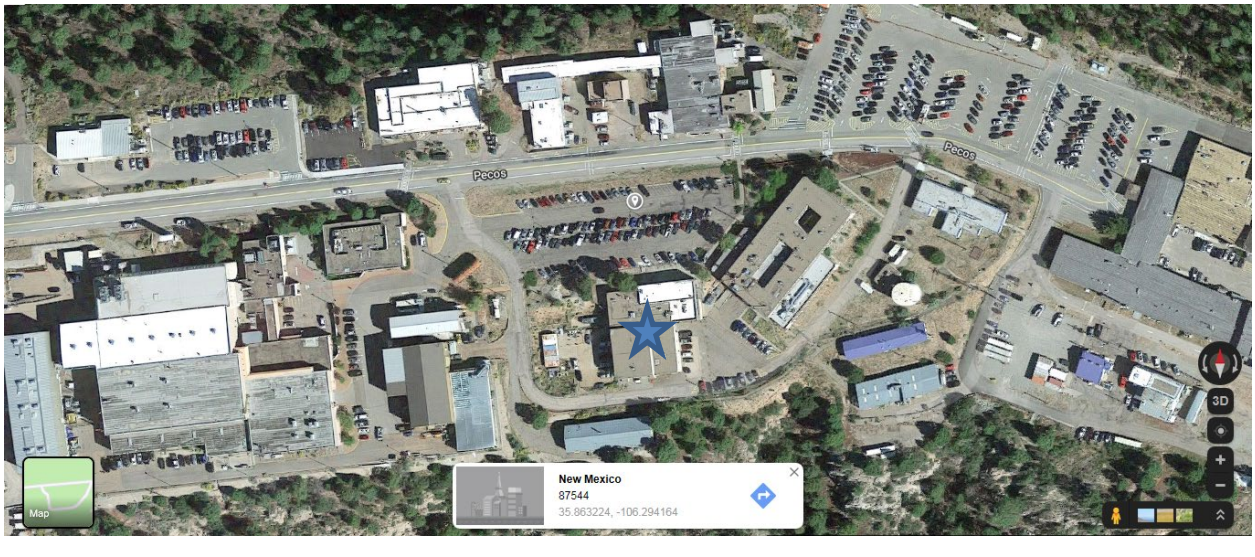
09:00 am: PF-4 Tour (TA-55 Access Center, Building 370)

09:00 am: RRFMC Tour (TA-03, Building 130, Room 103)

11:00 am: MINTS Demonstration (TA-03, Building 562)

01:00 pm: 2nd RRFMC Tour (if needed)

Building 86 in TA-35



Nuclear Material Packaging Program Review

Tuesday, August 20, 2019 Sign In Sheet

#	Signature	Name	Organization	email address	Clearance? (Q/L/U)	PF-4 Tour (7:30 8/21/19)	MINTS Tour (9:00 8/21/19)	RRFMC Tour (11:00 8/21/19)
1	[signature]	G.A BYINGTON	Y-12	JERRY.BYINGTON@	Q	✓		YES
2	[signature]			CNS.DOE.GOV				
3		LARRY HARMON						
4			PEL	LHARMON@PEL	NCT		✓	✓
5					BAC			
6	[signature]	William Gordon	NA-LA	william.gordon@nnsa.doe.gov	Q			
7	[signature]	CHARLES H. BROW	SRNL	chuck.brown@srs.gov	✓	✓	✓	✓
8	[signature]	CRAIG SIMON	Y-12	CRAIG.SIMON@CNS.DOE.GOV	Q	✓	✓	✓
9	[signature]	JOSEPH DUMONT	LANL	Joseph.DUMONT@lanl.gov	Q			
10	[signature]	Kwansoo Lee	C-CDE	kslee@lanl.gov	# U			
11	[signature]	Samantha Talley	LANL	sjtalley@lanl.gov	U			
12	[signature]	Andrea Labourian	LANL	andrea@lanl.gov	Q			
13	[signature]	Zachary Smith	LANL	zachary@lanl.gov	Q			
14	[signature]	Ray Vaidya	LANL	ray@lanl.gov	Q		✓	
15	[signature]	Jade Okda	LANL	okadj@lanl.gov	Q	✓		✓
16	[signature]	Tim Stone	LANL	stone_tim@lanl.gov	Q			
17	[signature]	John T. Davis	LANL	jdavis@lanl.gov	Q			
18	[signature]	Jonathan Gray	LANL	jgray@lanl.gov	U		✓	
19	[signature]	MURRAY E. MOORE	LANL	memoore@lanl.gov	Q			✓
20	[signature]	YONG TAO	LANL	yongtao@lanl.gov	Q			✓
21	[signature]	DUDUE JLAM	LANL	JDUDUE@LANL.GOV	Q			
22	[signature]	Daniel Rios	LANL	drios@lanl.gov	Q			
23	[signature]	ANDREW WYSONG	LLNL	WYSONG1@LLNL.GOV	Q			
24	[signature]	Noelani Laney	Y-12	Noelani.Laney@CNS.DOE.GOV	Q	✓		✓
25	[signature]	Robert Wingo	LANL	wingo@lanl.gov	Q			
27	[signature]	Danny Martinez	LANL	dmartinez@lanl.gov	Q			
28	[signature]	Matthew Fister	LANL	mwfister@lanl.gov	U			
29	[signature]	Robert Kanning	NNSS	KANNINRA@NV.DOE.GOV	Q	✓		✓
30	[signature]	BENJAMIN GARCIA	NNSS	garciaboenv.doe.gov	Q	✓		✓
31	[signature]	BEREKET KIFLU	NNSS	KIFLUBB@NV.DOE.GOV	Q	✓		✓
32	[signature]	MICHAEL BAKER	LANL	mcbaker@lanl.gov	Q			
33	[signature]	Tristan Karns	LANL	tkarns@lanl.gov	Q			
34	[signature]	Douglas K. Veirs	LANL	VEIRS@lanl.gov	Q			
35	[signature]	Joshua Narlesky	LANL	narlesky@lanl.gov	Q			
36	[signature]	Michael D. Heil	NA-LA	Michael.Heil@nnsa.doe.gov	Q			
37	[signature]	Laura A Worl	LANL	lworl@lanl.gov	Q			
38	[signature]	Cristy Abeyta	LANL	cabeyta@lanl.gov	Q			
39	[signature]	Paul Smith	LANL	smithp@lanl.gov	Q	✓		✓
40	[signature]	James Shaw	AU-32	james.shaw@hq.doe.gov	Q	✓		
	[signature]	David Grow	LANL	dgrow@lanl.gov	U			

on the phone (webex)

41		Bob Rosen	DNESB						
42		Richard Pierson	PMML						
43		Chris Chulupka	NNSS						
44		Nicole Schao	NNSA						
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Design Life Extension of the SAVY-4000 Series Containers

Timothy A. Stone, Kirk P. Reeves, Tristan Karns, Paul H. Smith, D. Kirk Veirs

19th International Symposium on the Packaging and Transportation of Radioactive Materials

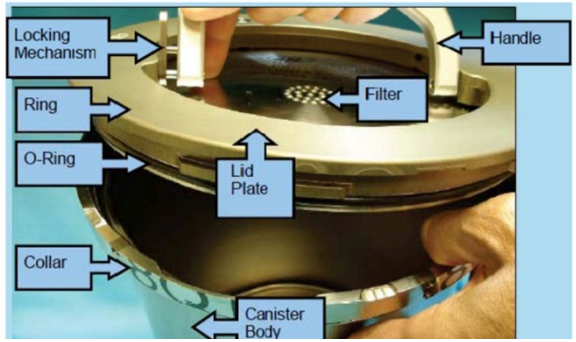
New Orleans, LA

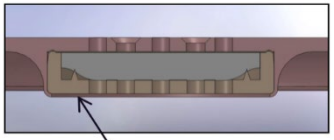
August 9, 2019

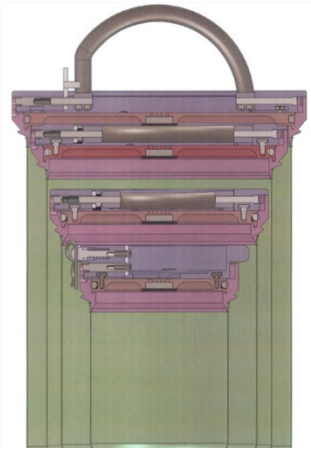
Presentation Outline

- Container Overview
  - Key Components
  - Initial 5 year design life
- Design Life Extension
  - Polymer Components
    - Viton O-ring
    - PTFE Membrane
  - Metal Components
    - Aluminum Locking Ring
    - Stainless Steel Body/Lid
- Future Plans

The SAVY-4000 Container Series

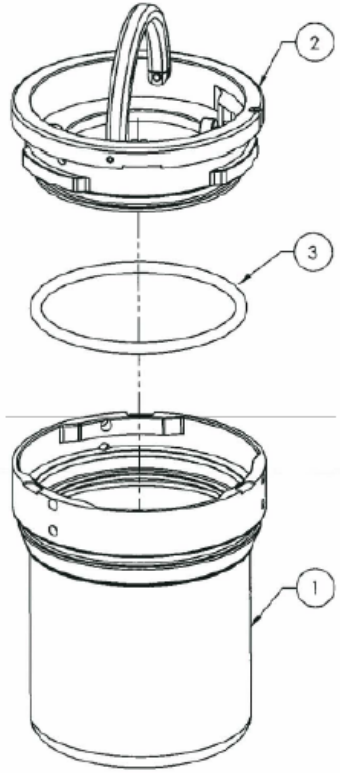






SAVY 4000 – M441.1-1 Compliant Container

- The body and lid are attached to one-another with a bayonet style closure
- The soft durometer Viton O-ring allows for opening/closing without tools and a water/air tight seal
- The lid locks into place with a positive mechanical engagement made of aluminum and a stainless steel pin
- The internal components that form the containment boundary are made of 316L stainless steel for corrosion resistance
- Containers designed to withstand a 12-ft drop
- Water-resistant containers
- Certified for MAR credit
  - "Cert" designation in LANMAS
  - Damage ratio: 0.01
- M441.1-1 requires design life justification
  - Initial Design Life = 5 years



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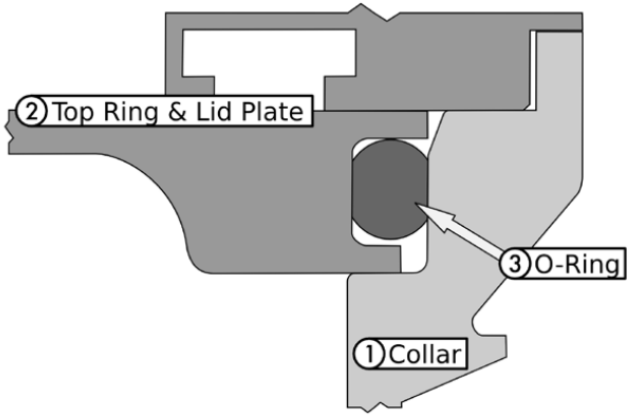

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Robust Manufacturing Surveillance Program

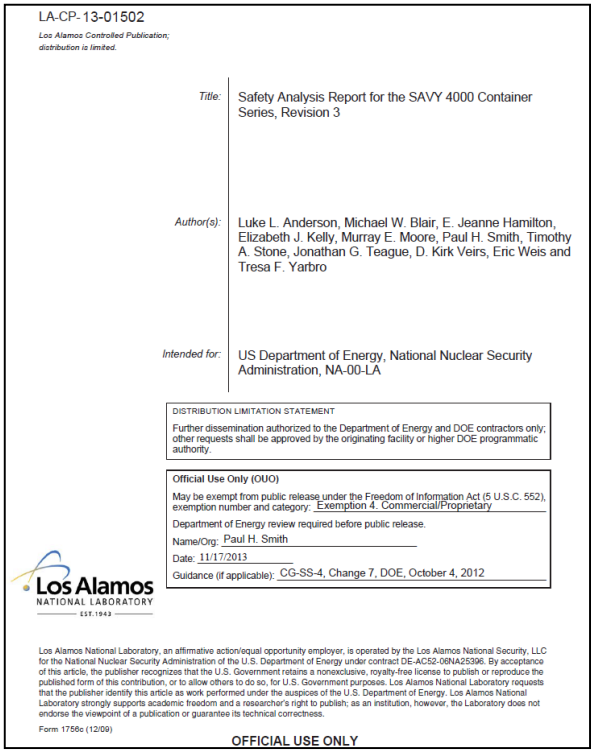
- 100% of SAVY containers are required to pass a helium leakage test at the time of manufacture
- 100% of SAVY containers are required to pass a filter test at the time of manufacture (efficiency, pressure drop, water penetration)
- 100% of SAVY containers are required to pass visual and dye penetration testing on the body/body and body/collar welds at the time of manufacture
- Additional random sampling (<3%) for destructive testing of body/collar welds is performed to show full weld penetration and solidification mode (primary ferrite and/or dual ferrite &austenite)
- 100% of SAVY lid and body O-ring grooves measured for surface finish at the time of manufacturing
- Additional 10% random sampling performed on He leak, filter, O-ring groove surface finish at QA Source Inspection (prior to shipping)

Hagan Containers – NOT M441.1-1 Compliant

- Threaded lid and body
- Closure action compresses Viton O-ring set in a groove above the body threads
- Internal components that form the containment boundary are made of 304L stainless steel
- Water resistant containers
- Certified for MAR credit
  - “Cert” designation in LANMAS
  - Damage ratio: 0.05
- Being phased out: no longer accepted byTA-55 vault for storage
- No design life established

Demonstrating SAVY-4000 M441.1-1 Compliance at LANL

- Revision 0 submitted January, 2011
- Q0 Comments received May, 2011
- Revision 1 submitted March, 2012
- Q1 Comments received January, 2013
- Revision 2 submitted March, 2013
- Q2 Comments received October, 2013
- Revision 3 submitted November, 2013
- OP&T issued SER, April, 2014
- LA Field Office Approved SAR, April, 2014



LA-CP-13-01502

Los Alamos Controlled Publication; distribution is limited.

Title: Safety Analysis Report for the SAVY 4000 Container Series, Revision 3

Author(s): Luke L. Anderson, Michael W. Blair, E. Jeanne Hamilton, Elizabeth J. Kelly, Murray E. Moore, Paul H. Smith, Timothy A. Stone, Jonathan G. Teague, D. Kirk Veirs, Eric Weis and Tresa F. Yarbro

Intended for: US Department of Energy, National Nuclear Security Administration, NA-00-LA

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Department of Energy review required before public release.

Name/Org: Paul H. Smith

Date: 11/17/2013

Guidance (if applicable): CG-SS-4, Change 7, DOE, October 4, 2012

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Form 1756e (12/09)

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Polymer Components: Accelerated Aging of Whole Containers

- 1-Qt. SAVY containers in each oven (70, 90, 120,160, 210°C)
- Periodically leak-tested. – No leaks
- Very difficult to remove lid after 9 months

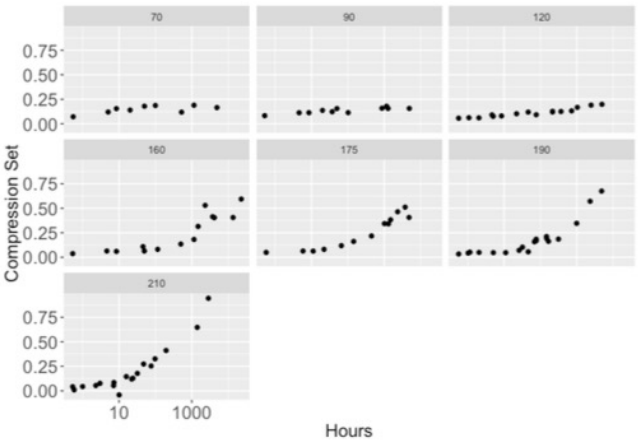
Accelerated Aging of Whole Containers to Determine Compression Set Failure Criteria

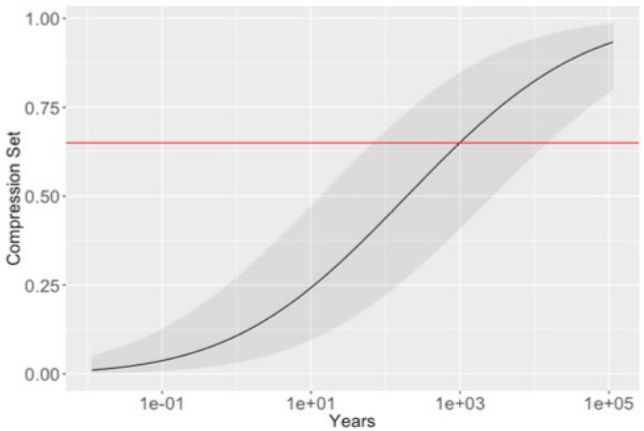
Aging Temperature	Compression Set	Leak Test Result Before O- ring Removal	Leak Test Result Immediately After O-ring Removal	Leak Test after O-ring Relaxation
210°C	93% ± 19%	Pass	Fail	Fail
160°C	13% ± 20%	Pass	Pass	NA
120°C	36% ± 14%	Pass	Pass	NA
90°C	18% ± 13%	Pass	Pass	NA
70°C	21% ± 8%	Pass	Pass	NA

- Failure point of O-ring in Compression Set → 65%

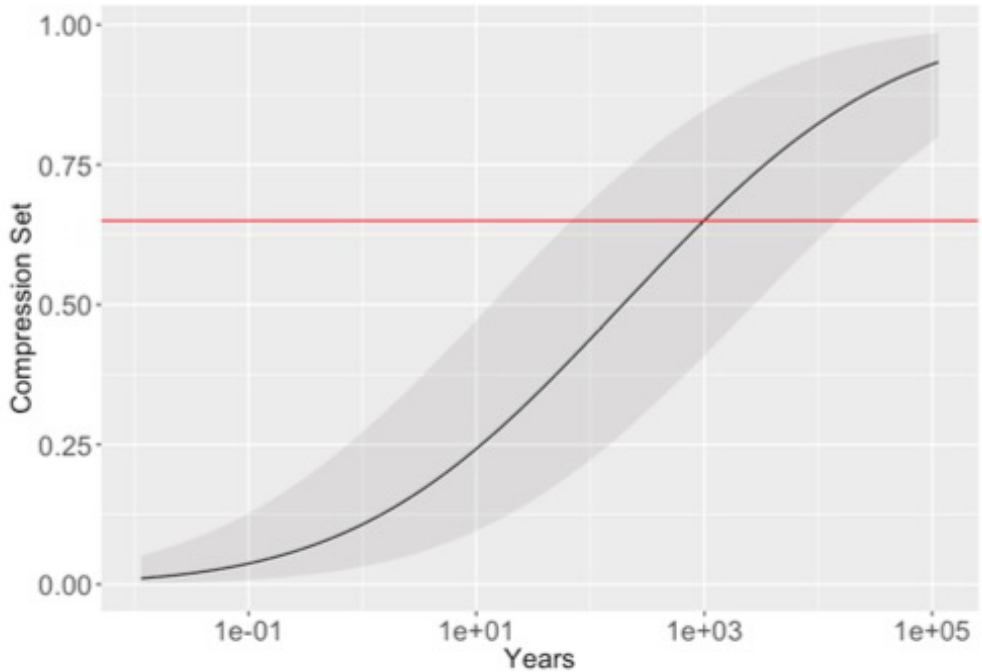
Compression Set and Statistical Model

- Compression Set experiments done to determine the amount of time that would elapse at the maximum service temperature of 80°C before the compression set attained would reach 65%
- Time temperature superposition proved inadequate for modeling the behavior of O-rings at low temperatures, predicting 267 to 39,675 years
- Statistical model was developed to incorporate all aging data





Compression Set and Statistical Model

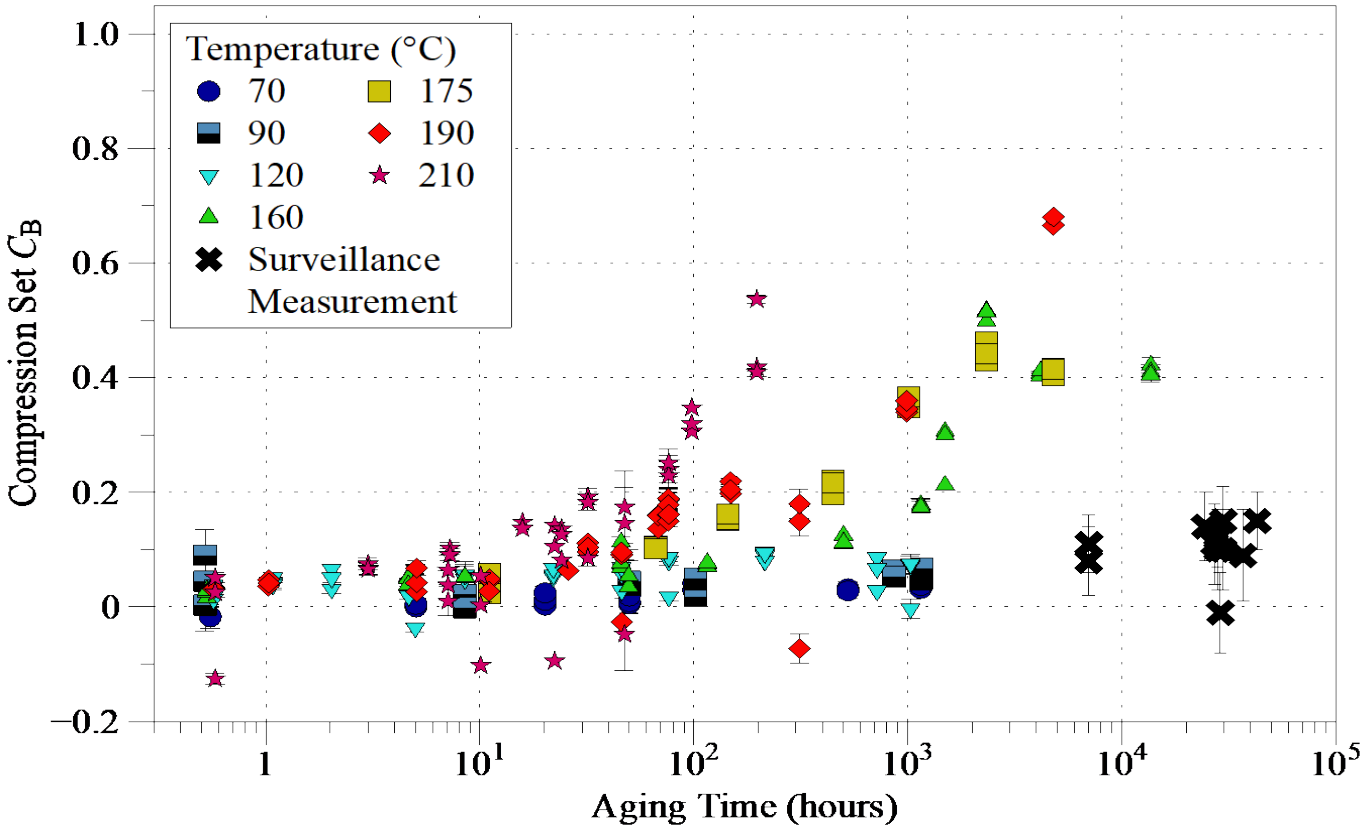


- Failure point of O-ring in Compression Set → 993 years
- Failure expected in the range of 68 to 15,000 years

Field Surveillance and Accelerated Aging Results are Consistent

Compression set on field samples is under 20%; these data follow the same trends exhibited by the low-temperature accelerated aging samples

Time-Temperature Superposition



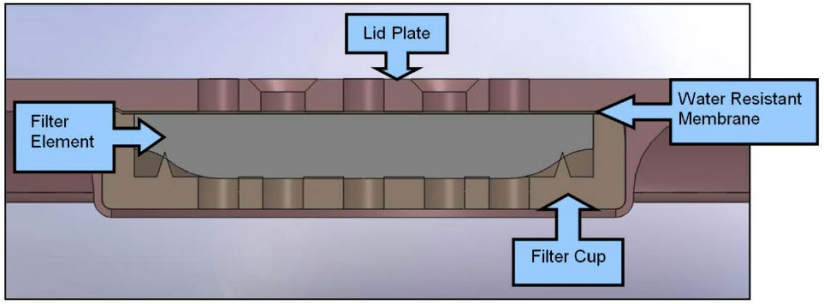
O-ring Design Life Conclusions

- Failure point of O-ring in Compression Set → 65%
- High confidence that 65% compression will not occur within 68 years
- Weak synergism between Heat and Radiation Dose
- Field Surveillance results consistent with accelerated aging results, thus far
- Desired lifetime of the o-ring is >40 years.

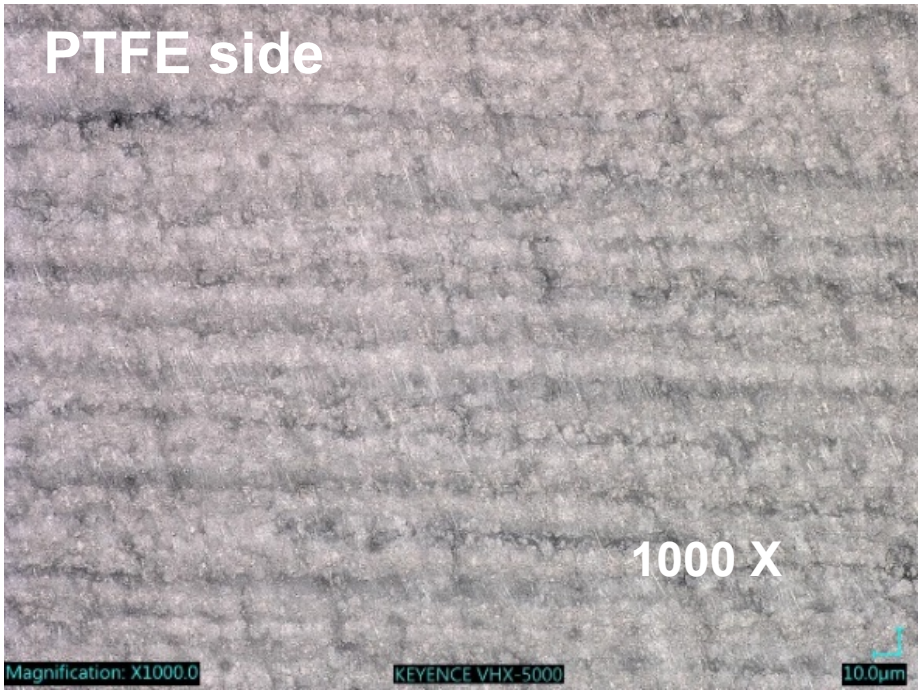
Filter Membrane Structure

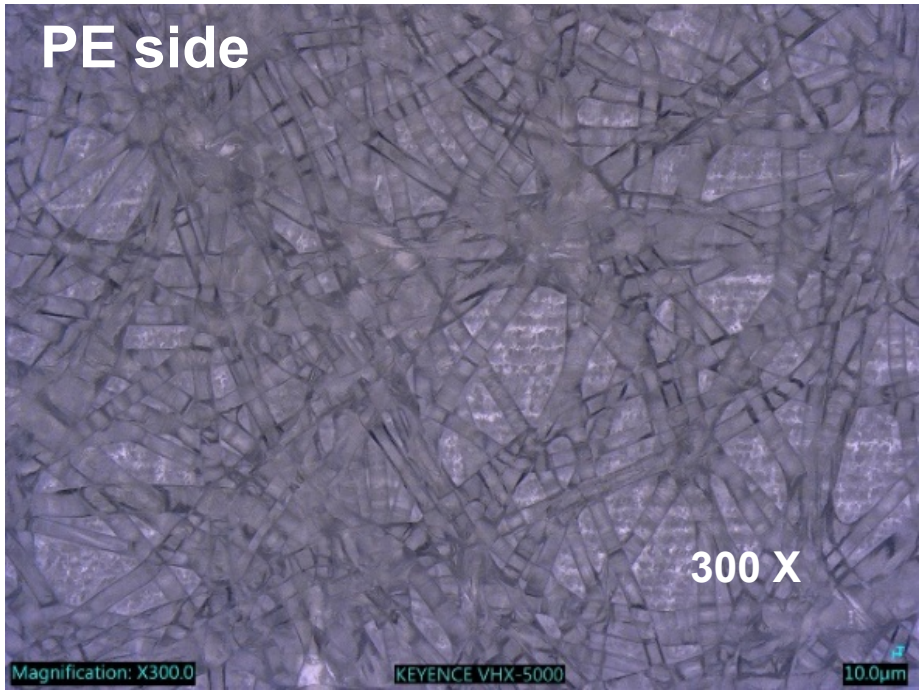
Digital Microscope Images

Water Resistance



Structural Support

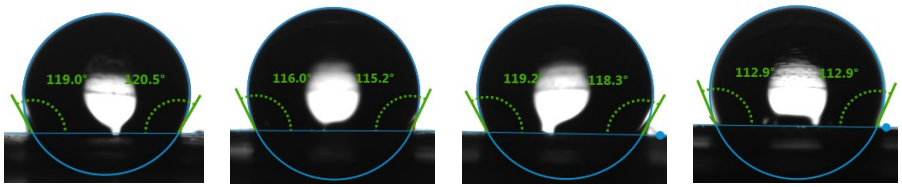




Membrane Exposure to γ Radiation

No Major Surface Changes → Maintains Hydrophobic/Waterproof Properties

- Polyethylene (PE) – Inner Side
  - Contact Angle
    - Slight Hydrophobicity Loss at Higher Doses



Pristine | 1 Mrad | 2 Mrad | 5 Mrad

Sample Exposure Details

Modeled Exposure Time	Average Contact Angle (Degrees)
Pristine	120
1 Mrad	116
2 Mrad	118
5 Mrad	112

- Teflon (PTFE) – "Waterproof" Outer Side
  - Contact Angle
    - Mostly Maintains Hydrophobicity

Pristine | 1 Mrad | 2 Mrad | 5 Mrad

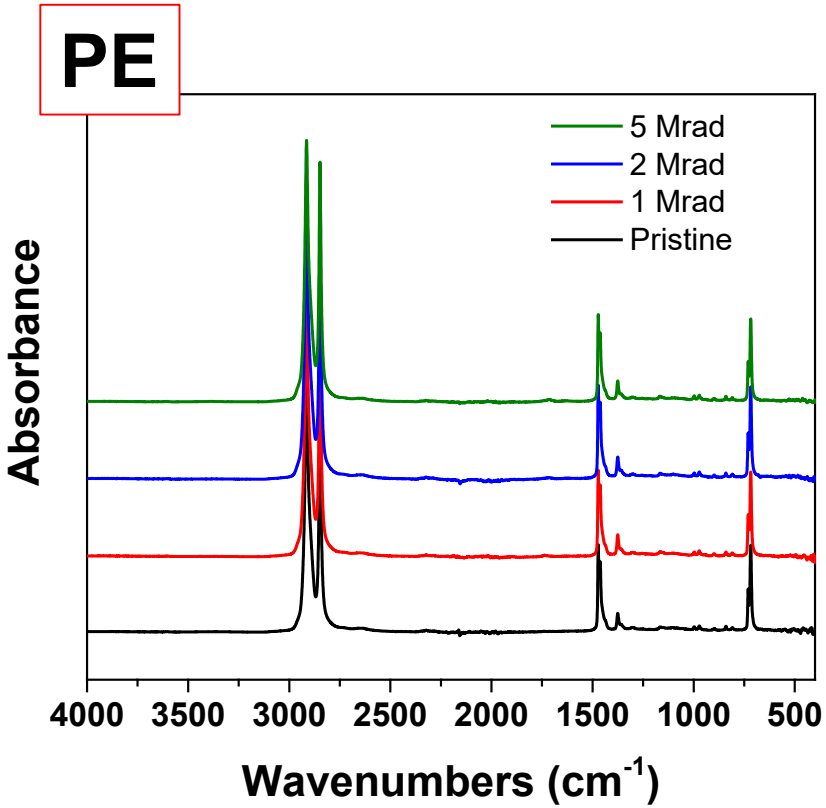
Sample Exposure Details

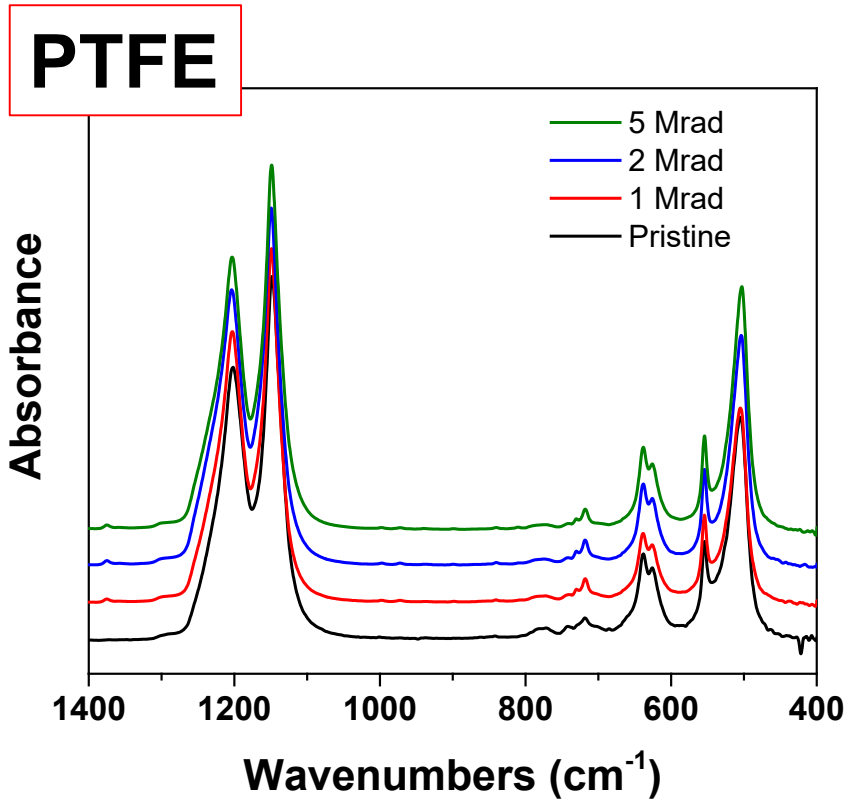
Modeled Exposure Time	Average Contact Angle (Degrees)
Pristine	140
1 Mrad	142
2 Mrad	146
5 Mrad	140

- ***Note: Membranes are expected to receive an estimated dose of 4.2 Mrad over 40 years.***

Membrane Exposure to γ Radiation

FTIR Shows No Significant Chemical Changes



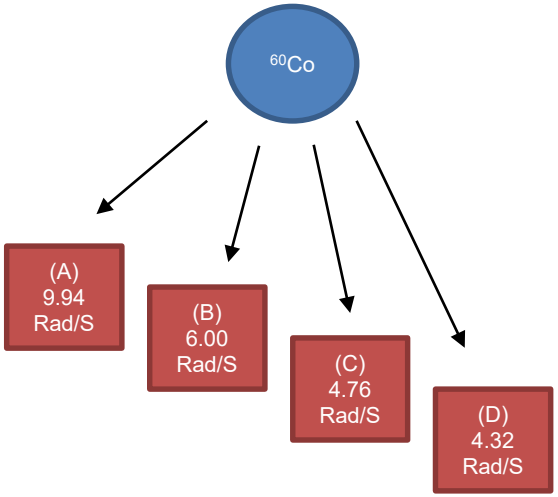


→ No substantial chemical degradation observed after 5 Mrad gamma exposure.

Gamma Irradiation Facility Experiment

- Four Whole 1 Quart SAVY-4000® containers laying on side with filter pointing toward Cobalt-60 source in furnace, one in each furnace
- All four SAVY-4000® containers passed water penetration test (12 inch WC, 1 minute) after Round 3
- All four SAVY-4000® containers failed water penetration test (12 inch WC, 1 minute) after Round 4

- Round 1 (Room Temp, 72 Hours)
  - A – 2.58 Mrad
  - B – 1.56 Mrad
  - C – 1.23 Mrad
  - D – 1.12 Mrad
- Round 2 (70ºC, 72 Hours)
  - A – 5.16 Mrad
  - B – 3.12 Mrad
  - C – 2.46 Mrad
  - D – 2.24 Mrad

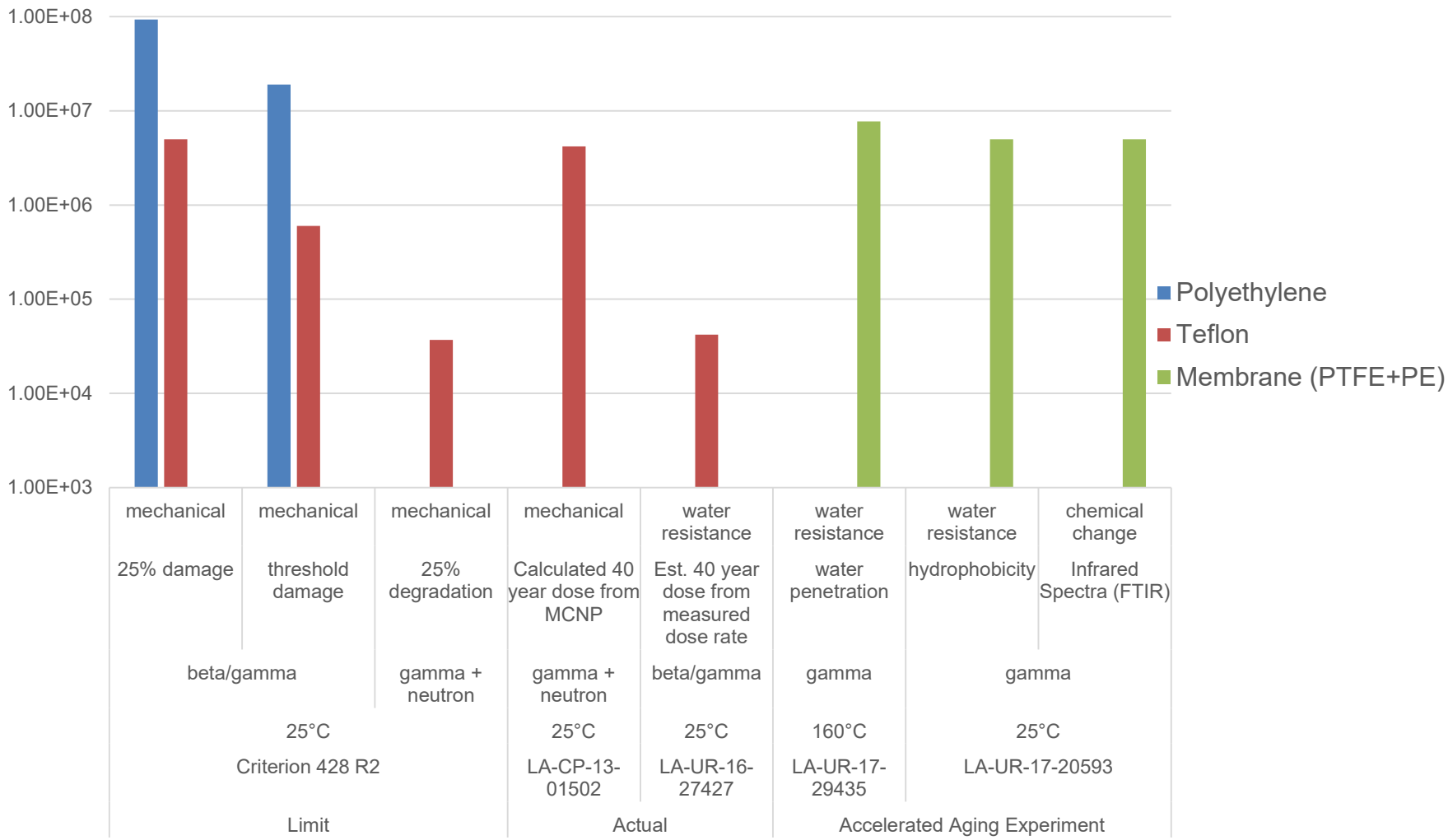


- Round 3 (160ºC, 72 Hours)
  - A – 7.74 Mrad
  - B – 4.68 Mrad
  - C – 3.69 Mrad
  - D – 3.36 Mrad
- Round 4 (210ºC, 72 Hours)
  - A – 10.32 Mrad
  - B – 6.24 Mrad
  - C – 4.92 Mrad
  - D – 4.48 Mrad

Membrane Design Life

- The calculated dose over 40 years (4.2 Mrad) far exceeds what will actually be seen – this is supported by actual dose measurements of materials chosen for surveillance (4.2×10^4 rad).
- At 5×10^6 rad beta/gamma PTFE/PE membrane (actual container component) maintains hydrophobicity
- Whole SAVY lid experiments at high gamma dose rates and elevated temperature demonstrate 7.7×10^6 rad at 160°C
- Surveillance includes water penetration testing and will continue

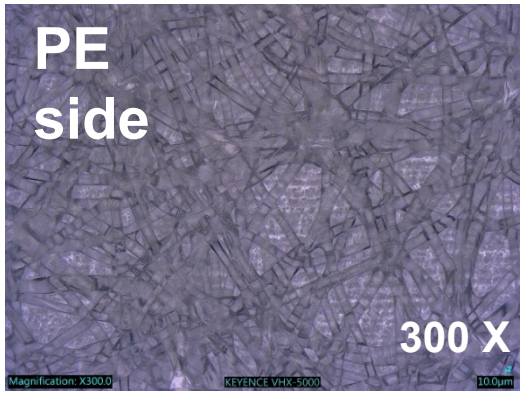
PTFE/PE Membrane Dose

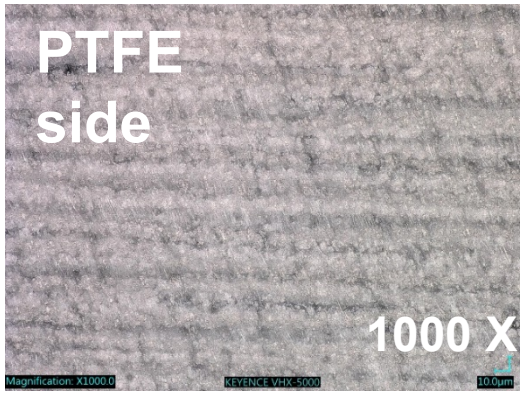


Membrane Degradation by α Radiolysis

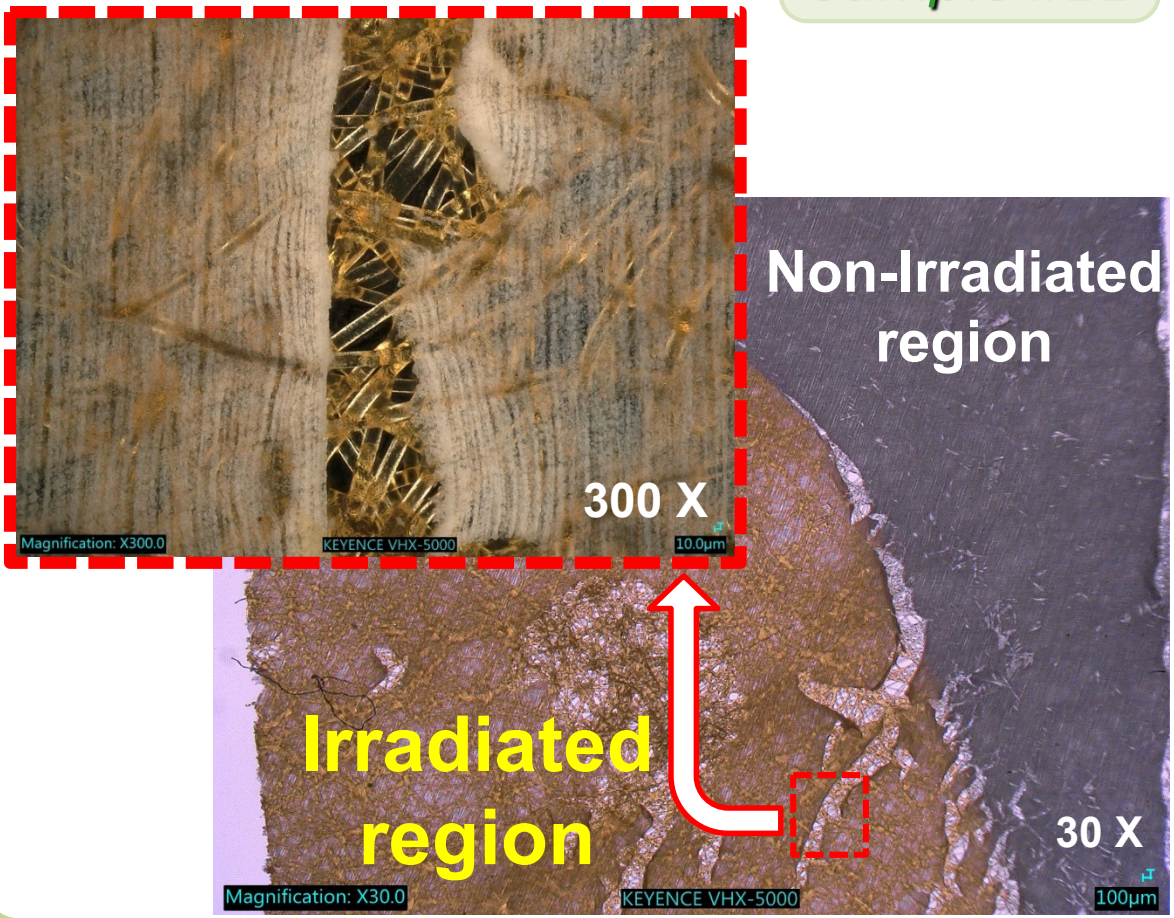
Digital Microscope Images Reveal Extensive Structural Damage

Pristine





Sample #11



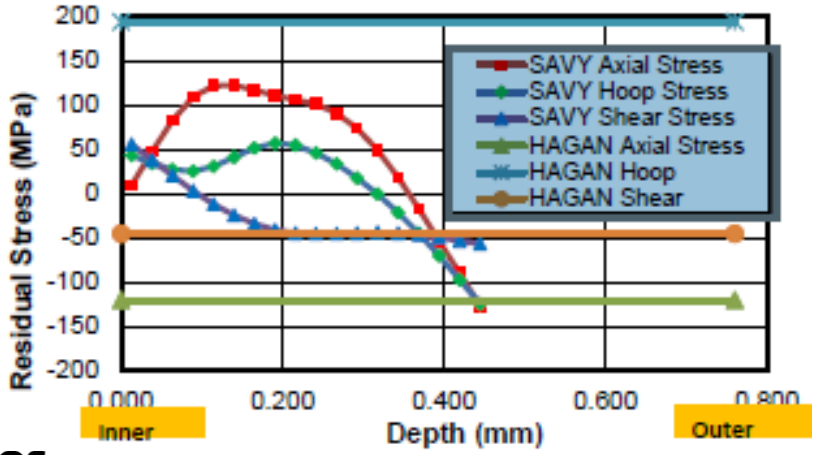
→ *Sample #11 (5e13 α/cm^2 , 1e10 α/cm^2/sec) showed cracks and discoloration.*

Stress Corrosion Cracking Susceptibility

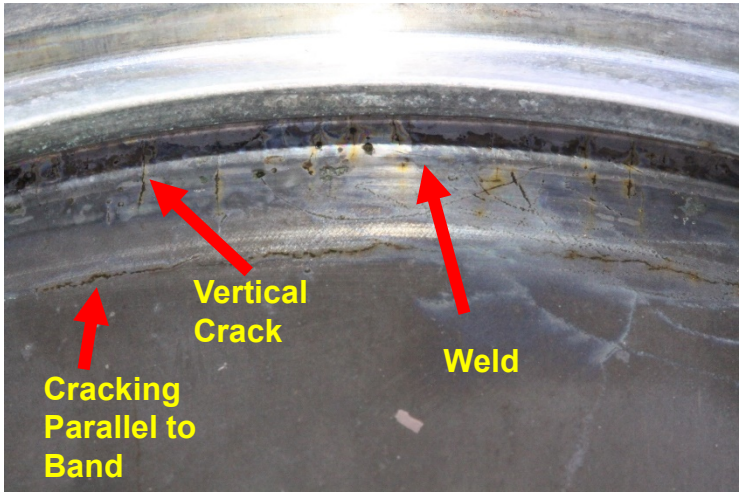
Boiling $MgCl_2$ testing

- Provide evidence that the SAVY-4000 is more robust than the Hagan
- Use historic data from materials stored in Hagans to extend the approved lifetime of the SAVY-4000 container beyond 5 years

- Average tensile hoop stresses
  - Hagan: 194 MPa
  - SAVY: 25 MPa



SAVY-4000 (44-46 hours)



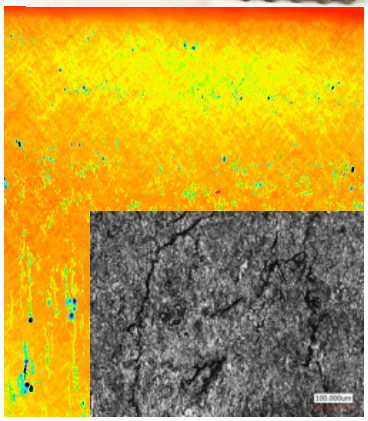
Cracks not through wall

Hagan (22-24 hours)

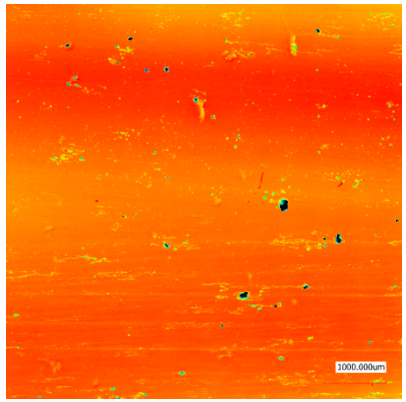
Laboratory Corrosion Studies

- Accelerated corrosion studies
- Exposure to 6M HCl vapor up to 168 days
- Specimens
  - Pitting observed in both experiments
  - Corrosion density increases with exposure time
  - Cracking observed on Hagan specimen
- Containers
  - SAVY-4000 failed leak test due to pin hole in wall
  - Hagan: TBD

126 Days, 59% RH



126 Days, 61% RH



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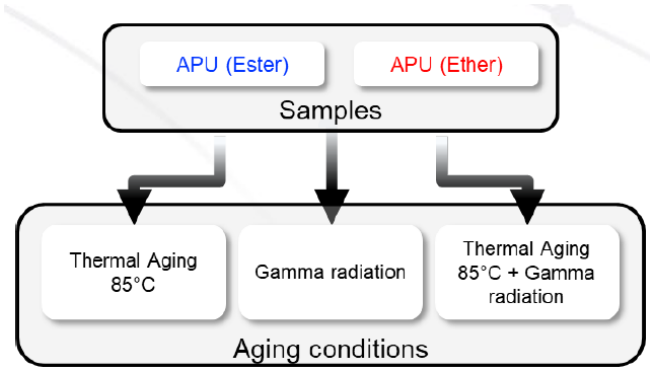
NNSA National Nuclear Security Administration

Corrosion: can justify lifetime extension to 15 years

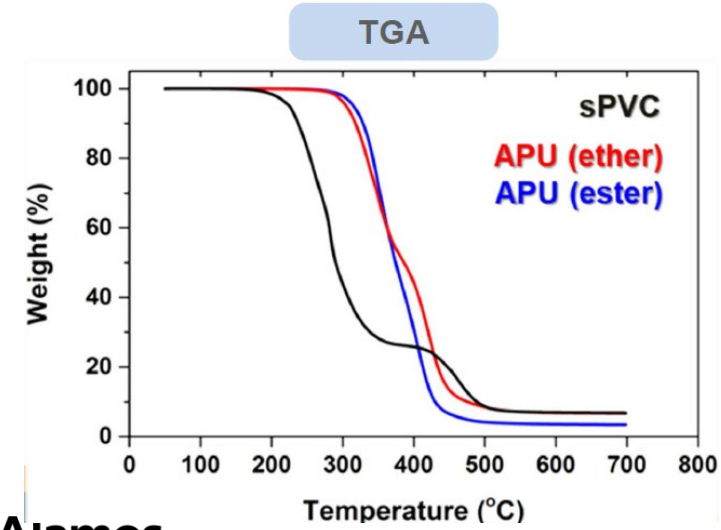
- General corrosion
  - Worst general corrosion in a SAVY-4000 was in the SAI container
    - Reduction in sidewall thickness of SAI SAVY container was negligible
    - SAI SAVY container passed He leak test after 4 consecutive drop tests
  - Worst general corrosion in Hagan was in the “Stuck Hagan”
- Pitting
  - Deepest pits in SAVY-4000: SAI SAVY was ~60 µm (1 year packaging exception)
  - Deepest pits in Hagans: “White-Powdered Hagan”: 35 µm deep (8 years) and “Stuck Hagan” ~ 40 µm deep (13 years)
  - Pitting won’t challenge container within 40 year lifetime
- SCC Susceptibility
  - SAVY-4000:
    - No thru-wall cracks in SAVY-4000 containers (by visual inspection and He leak test)
    - Cracks of ~30 µm in length emanating from bottom of pits in SAI SAVY (1 year packaging exception)
  - SAI SAVY passed He leak test, no thru-wall cracks observed by visual inspection
  - Hagans: No cracking observed by visual inspection (age 16 years)
  - Hagans more likely to crack based on boiling $MgCl_2$ test

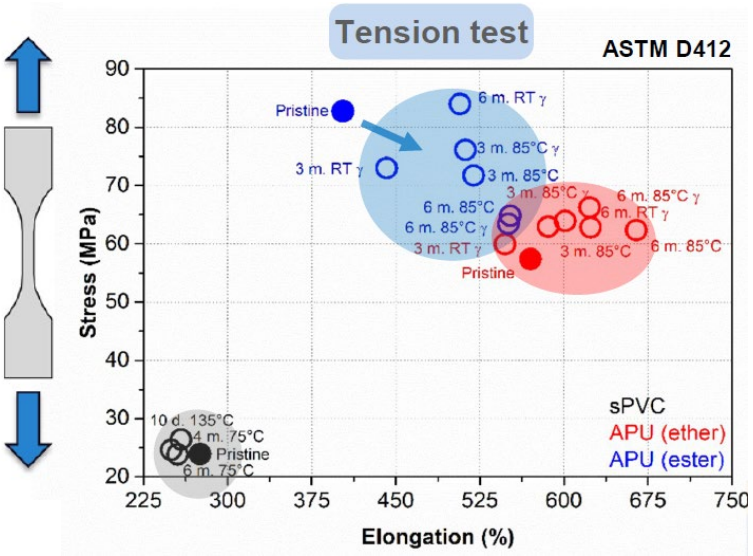
PVC Bag Replacement

- Bags for the storage of nuclear materials are crucial for TA-55
- Aromatic polyurethane (APU) alternatives were selected and aged to reproduce real-life conditions
- APU shows excellent thermal, mechanical, and chemical stability after 6 months of accelerated aging



Samples	Toughness (MPa)
sPVC (pristine)	10.7
sPVC (aged)	5.1
APU (pristine)	**20.7**
APU (aged)	**19.2**





Joseph H. Dumont
Los Alamos National Laboratory

Clive Young Award for Early Career Outstanding Poster Presentation

August 8, 2019

"Degradation of sPVC and Aromatic Polyether Urethane Bags Used in Nuclear Applications"

Future Plans

- Surveillance
  - Targeting worst case items
  - Add dye penetration testing to surveillance activities
  - In GB He leak testing of Hagan containers
  - Target SAVY-4000 containers for lid bar stock issue
- Cold studies of HCl vapor corrosion of Hagan/SAVY-4000 containers
- Reduce potential for corrosion in future packaging by replacing PVC bags

DOE/NNSA Los Alamos Field Office Approval

- On June 11, 2019 the DOE/NNSA Los Alamos Field Office (NA-LA) sent a letter to the Triad Prime Contract Office Manager approving the design life extension for the SAVY-4000 Container Series from 5 Years to 15 Years.
- The 316L stainless steel wall is the life limiting component. All other components could be extended to >40 years based on accelerated aging results.
- The design life extension can be made available to other DOE sites to consider approving the changes at those sites for material that is packaged within the boundaries of the defined content envelope for the SAVY.

Acknowledgements

Radiation Protection:	Murray Moore, Yong Tao
Materials Science:	Eric Weis, Cynthia Welch, Matthew Herman, Mary Ann Hill, Justin Tokash
Chemistry:	Andrea Labouriau, Joseph Dumont, Anna Carroll, Kwan-soo Lee, Juan Duque
Engineering:	Joshua Narlesky, Daniel Rios, John Berg, Mary Ann Stroud, Kirk Veirs, Laura Worl, Jude Oka, John Davis, Vera Aguino
Statistical Science:	Elizabeth Kelly, Kim Kaufeld, Brian Weaver

SAVY/Hagan Corrosion Update

Joshua Narlesky,
John Davis, Tristan Karns, Kim Kaufeld, Elizabeth Kelly, Jude Oka, Kirk Reeves, Daniel Rios, Paul Smith, Tim Stone, Mary Ann Stroud, Ed Romero, Kennard Wilson

August 20, 2019

Managed by Triad National Security, LLC for the U.S. Department of Energy's NNSA

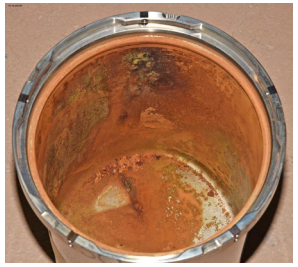
Introduction

Typical storage configuration

- **10-year life extension for SAVY containers approved**
- **Cause of corrosion on storage containers is known**
  - HCl gas is produced from the radiolytic (and thermal) decomposition of PVC bag
  - HCl gas can cause general corrosion, pitting, and stress corrosion cracking (SCC) in 304L and 316L stainless steels
  - Production of HCl gas increases with increasing beta/gamma dose from the packaged material
- **Assess impact of corrosion through surveillance program**
  - Use engineering judgement to select most aggressive storage conditions in Hagan and SAVY containers
  - Include Hagan containers packaged for up to 18 years to understand aging
  - Storage conditions for Hagans expected to be bounding
    - Contents (material types and forms)
    - Age (> 5 years)
    - Hagan containers more susceptible to SCC, based on boiling magnesium chloride testing and residual stress measurements

Solution Assay Instrument (SAI) Standards Containers

- **Material Characteristics:**
  - Pu in 3M HCl solution in plastic volumetric flasks with individually placed inside taped PE bags.
- **Storage Conditions (1.5 yr):**
  - HCl gas / high relative humidity
  - 10 ppm HCl / ~ 80% RH
- **Inspection**
  - Corrosion product covering inside surface and collecting at bottom.
  - Corrosion did not pass O-ring.
- **Analysis**
  - Analytical chemistry: NH_4Cl and SS corrosion product
  - Wall thickness: Coordinate measuring machine and ultrasonic testing found no significant change
  - Drop Test: Passed He leak test following four consecutive drops (average 1.65E-08 atm-cc/s)
  - Laser Confocal Microscopy (Juan Duque)
  - Accelerated aging studies (Daniel Rios)

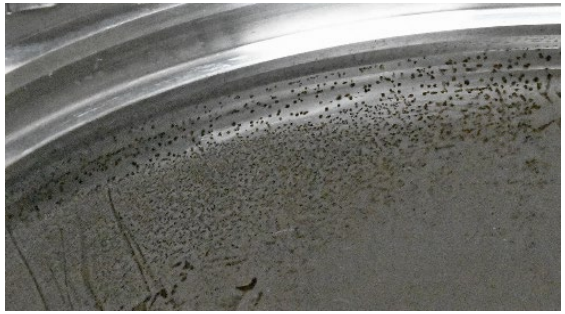


FY18 Surveillances

- More emphasis on corrosion due to PVC degradation:
  - High Wattage/High Am
  - Older Hagans
- Limitation due to 500 g restriction on handling soluble residues lifted.
- SAVY Containers: 7
  - Age: 1 to 5.6 years
  - All passed performance criteria
  - Minor corrosion on 4 containers
  - Moderate corrosion on 1 container
- Hagan Containers: 8
  - 6 to 17 years
  - Corrosion on 4 containers
  - One fused lid (corrosion past O-ring sealing surface)

BLO-39-11-16, Pu Oxide, 5 W

2017

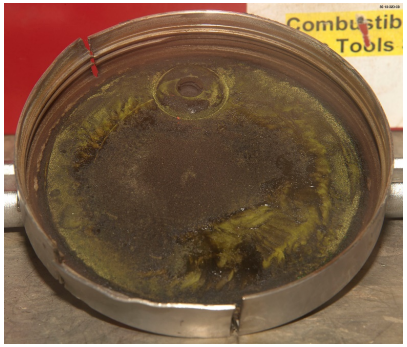


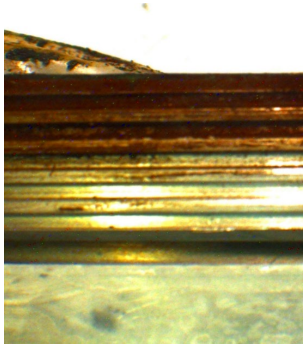
2018

18H4, MSE Salt, 0.45 W, 6.2 years

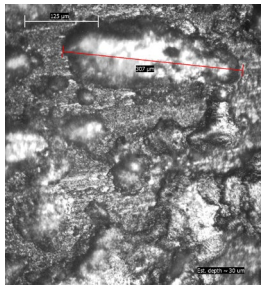
18H7, MSE Salt, 6.9 W, 13 years Fused Lid

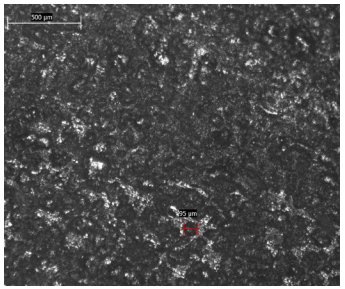
“Stuck” Hagan

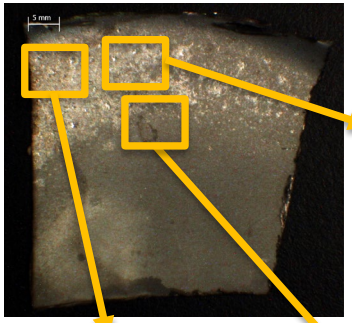


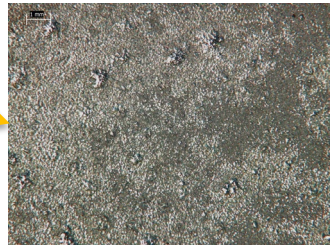


- **Worst corrosion to date**
- **MSE Salt, 6.9 W, 451 g Pu**
  - Stored 13 years
  - Configuration: (a) taped slip lid inside (b) one bagout bag inside (c) 1-Qt Hagan container
- **Corrosion past sealing surface—lid fused to body due to corroded threads**
- **Wet coating, green to brown in color throughout container**
- **Microscopic examination**
  - Difficult to find native surface due to general corrosion
  - No evidence of cracking
  - **Lid:** large diameter pits (100-150 μm) ~ 20 to 40 μm deep (est.)
  - **Side wall:** general corrosion
  - **Bottom:** Large eroded areas





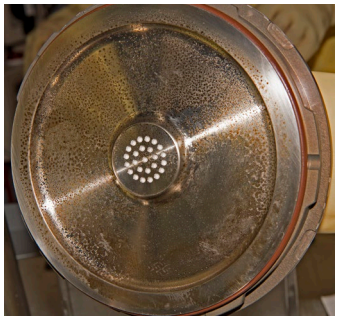




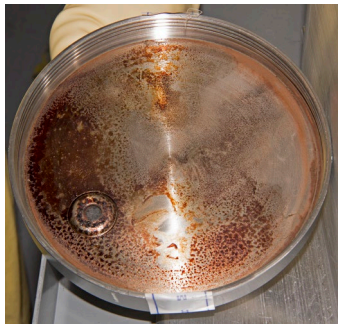
FY19 Surveillances

- Continued emphasis on corrosion due to PVC degradation
  - Old Hagans with high wattage, high Am, smaller containers
  - Same IDCs from FY16-18
- Oldest SAVYs with > 500 g Pu
- SAVY Containers: 7
  - Age: 2.6 to 6.7 years
  - Minor corrosion on 4 containers
  - Moderate corrosion on 1 container
- Hagan Containers: 8
  - 10.8 to 18.1 years
  - Corrosion on 6 containers
  - Two containers had corrosion outside the O-ring sealing surface
  - One fused lid

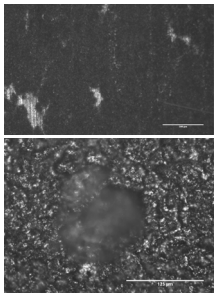
19S13, Oxide High Pu-240, 2,059 g Pu, 10.5 W, 3.5 yr, 5Qt

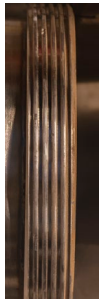


19H1, Pu-242 Metal, 2,165 g Pu, 14.9 W, 18.1 yr, 3Qt



19H5, MSE Salt, 356 g Pu, 5.7 W, 12.5 yr, 1Qt



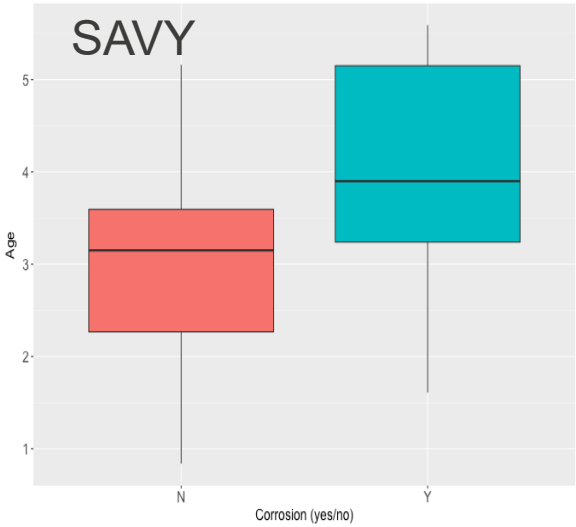


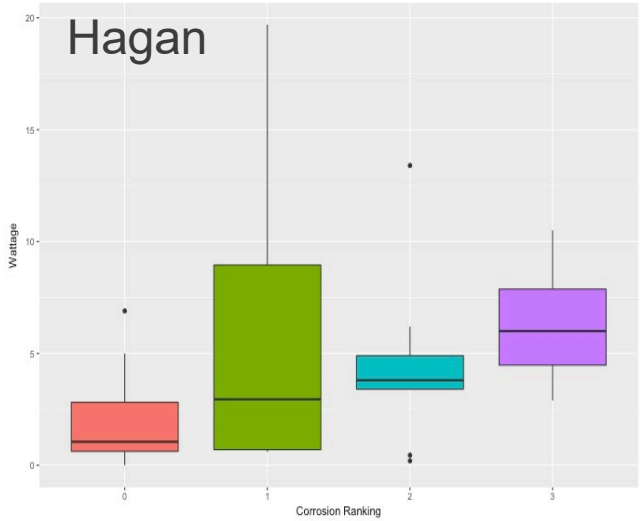
Storage Container Summary

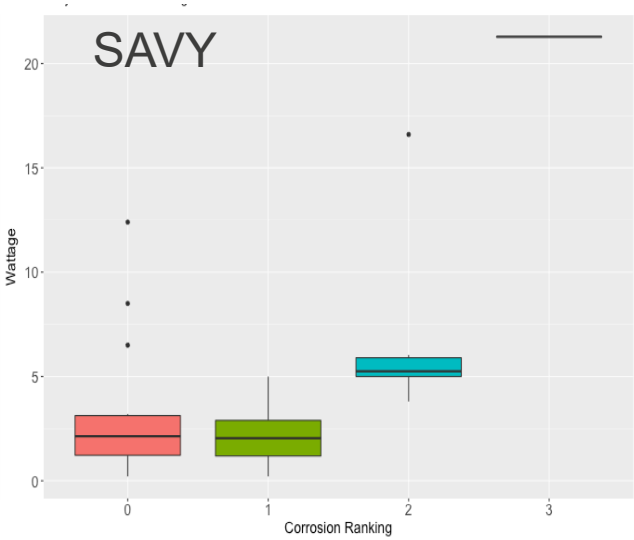
- **SAVY containers: 17 of 47 containers (1.6 - 6.7 years) had corrosion**
  - Corrosion observed outside the O-ring sealing surface: 0
- **Hagan containers: 25 of 43 containers (5.6 to 18.1 years) had corrosion**
  - Corrosion observed outside the O-ring sealing surface: 3
- **Microscopic examination performed on 3 Hagan containers**
  - General corrosion and pitting observed
  - Pit sizes vary widely.
    - Individual pits 10-50 microns in diameter.
    - Agglomeration leads to pits 100-200 microns in diameter
    - Maximum depth estimated to be ~40 microns (shortest duration—8 yr)
- **Helium leak testing has not indicated any through wall penetration**

Corrosion Ranking

	No. Containers SAVY | Hagan	Description	Criteria
0	30 | 18	No Corrosion	No corrosion, staining, spots, or coatings observed
1	8 | 7	Isolated General Corrosion	Corrosion, staining, spots, or coatings observed in isolated areas (e.g. corrosion found on weld only)
2	8 | 11	Light General Corrosion	Corrosion, staining, spots, or coatings throughout container; light in overall density; bare metal visible
3	1 | 7	Heavy General Corrosion	Corrosion, staining, spots, or coatings throughout container; heavy (dark) in overall density; little or no bare metal visible







Factors Affecting Corrosion

- **Material form**
  - MSE Salts: 11 of 21 have corrosion
- **High specific wattage**
  - Pu-238: 100% have corrosion when PVC bag is present
  - High Pu-240 (and Am-241): 18/28 had corrosion
- **Age > 10 years: 26 of 45 Hagans have corrosion**
- **Container size**
- **Bag degradation factor:**
  (Age*Wattage/Radius^2)
- **<u>However,</u> identical items with different behavior suggest that environmental factors must be considered**
  - Location
    - Temperature of storage location
    - Relative humidity of storage location
  - Package
    - Inner and/or outer shielding
    - PVC bag composition

ITEM700—Pu metal
<4% Pu-240, 679 g Pu, 1.5 W, 17.5 yr, 5Qt

ITEM800—Pu metal
<4% Pu-240, 674 g Pu, 1.5 W, 17.5 yr, 5Qt

Path Forward

- **Continue to target most aggressive storage conditions in surveillance to support life extension**
  - Continue to monitor SAVY containers known to have corrosion
  - Perform surveillance on SAVY containers with packages from corroded Hagan containers
- **LCM analysis of Hagan container specimens**
- **Nondestructive testing system to measure wall thickness and detect defects in surveillance**
- **Accelerated aging studies to understand the susceptibility of Hagan and SAVY containers to stress corrosion cracking (and pitting) under extreme conditions**
- **Remove source of HCl through PVC bag replacement**

Accelerated Corrosion Experiments with SAVY and Hagan Containers

Los Alamos National Laboratory

Daniel Rios, Jim Wendelberger, Juan G. Duque

Authors Contribution

***Rios:** Designed and performed experiments. Helped with report and data interpretations.

***Wendelberger:** Developed Matlab software.

***Duque:** Collected and analyzed all data. Ensemble most of the report and perform data interpretation

Special thanks to LANL SAVY corrosion working group for helpful discussions: Paul Smith, Kirk Reeves, Tristan Karns, Timothy Stone, John Berg, Josh Narlesky, Elizabeth Kelly, Kimberly Kaufeld

Purpose for Performing these Experiments

- SAI Containers (two):
  - $PuCl_3$ in 150 mL of 3M HCl
  - ~1.5 years in storage
- SAI container passed leak test after several drops
- SAI container has pits with no cracks and pits with cracks

Can we identify accelerated conditions that would lead to this corrosion?

Experimental Conditions

SAVY

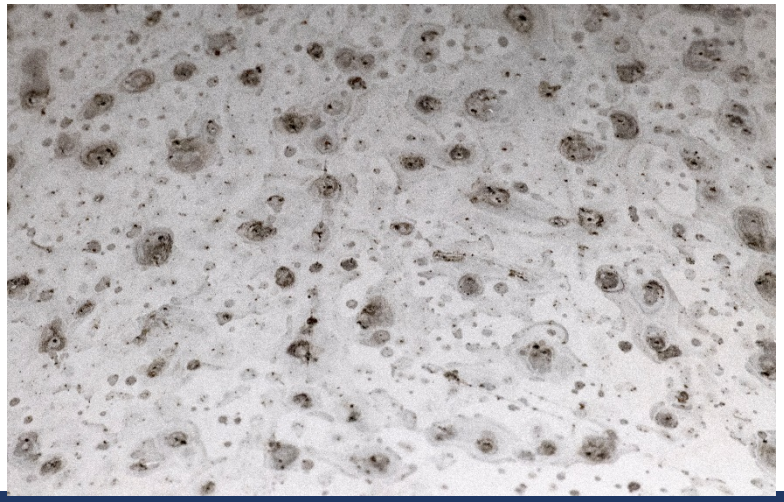
- 150 mL of 6M HCl in a 250 mL Erlenmeyer flask. Flask open.
- Four corrosion specimens
- RH reached equilibrium at ~ 66%
- Initial HCl concentration at the neck of the flask was 48 ppm
- Close system

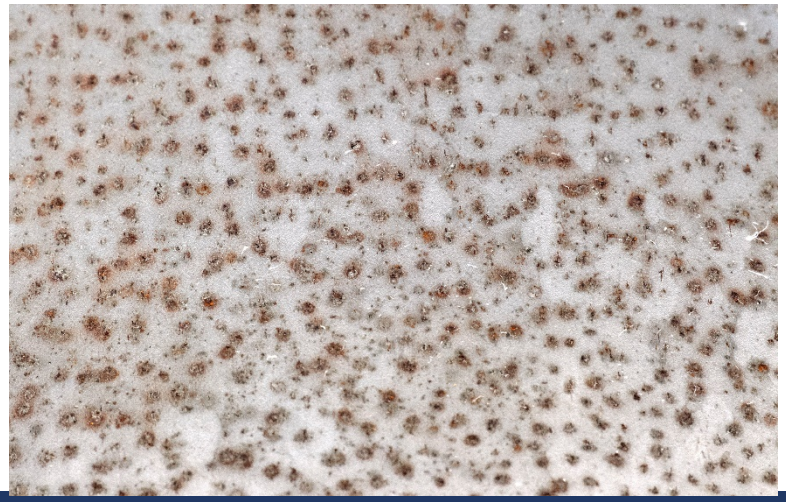
Hagan

- 150 mL of 6M HCl in a 250 mL Erlenmeyer flask. Flask open.
- Four corrosion specimens
- RH reached equilibrium ~ 62 %
- Initial HCl concentration at the neck of the flask was 48 ppm
- Close system

Corrosion in SAVY and Hagan Containers After Exposure to 6M HCl for 31 Days

Appearance in SAVY and Hagan Containers After Corrosion Removal





Corrosion on SAVY Specimen Exposed to 6M HCl Vapors

31 Days, 65% RH

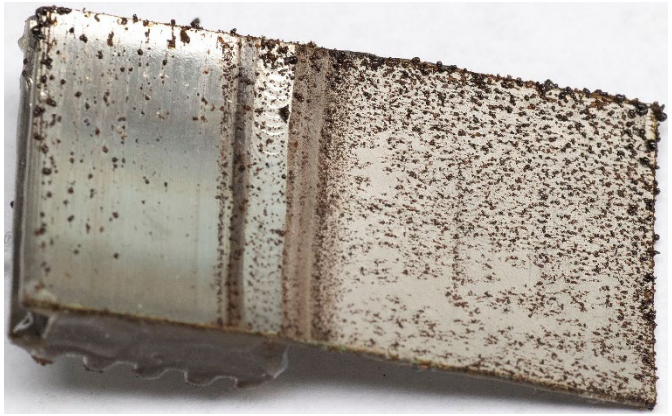
63 Days, 64% RH

95 Days, 61% RH

126 Days, 61% RH

Corrosion on Hagan Specimens Exposed to 6M HCl Vapors

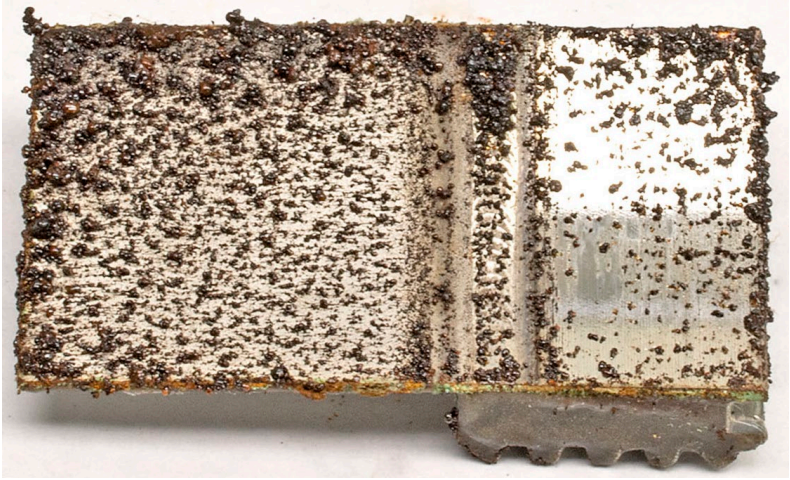
31 Days, 62% RH



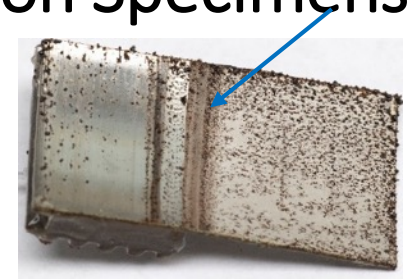
63 Days, 62% RH

95 Days, 60% RH

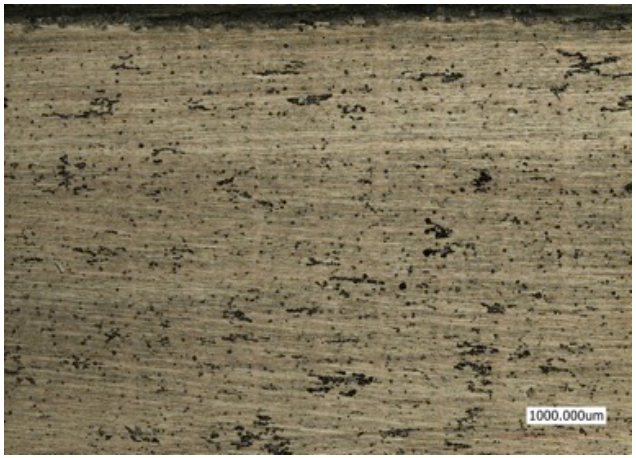
126 Days, 59% RH



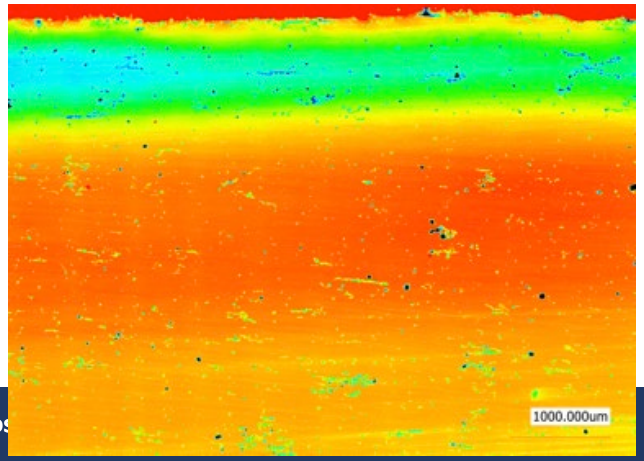
LCM Analyses on Corrosion Specimens



Stitched image (10x10)

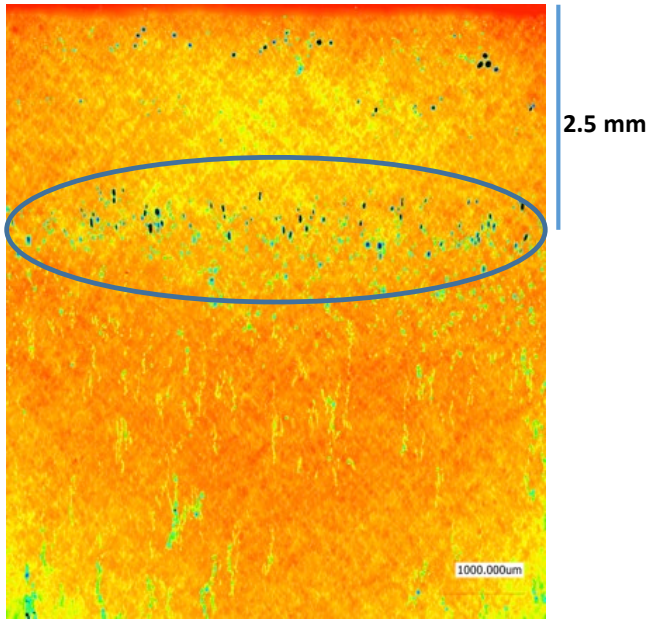


Height image



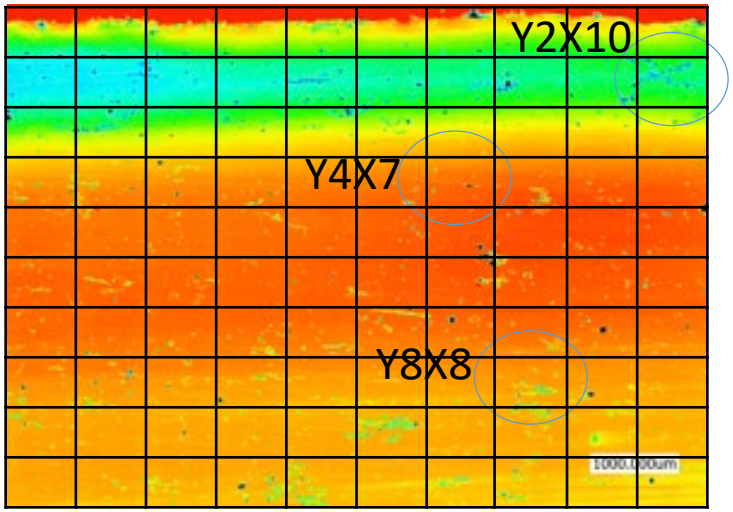
Stitched image (10x16)

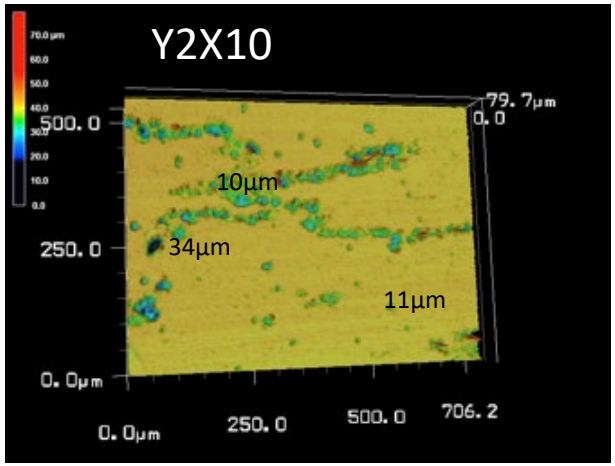
Height image

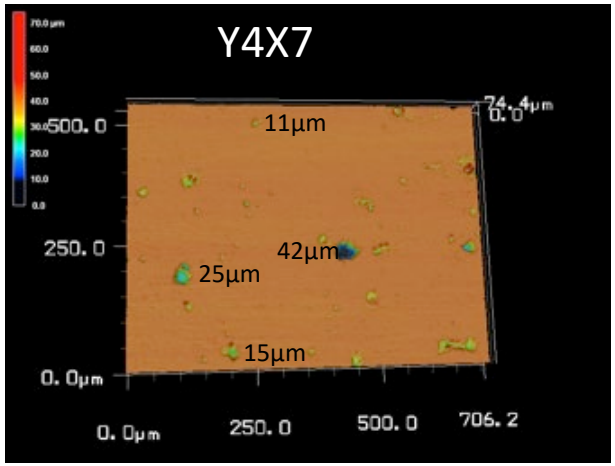


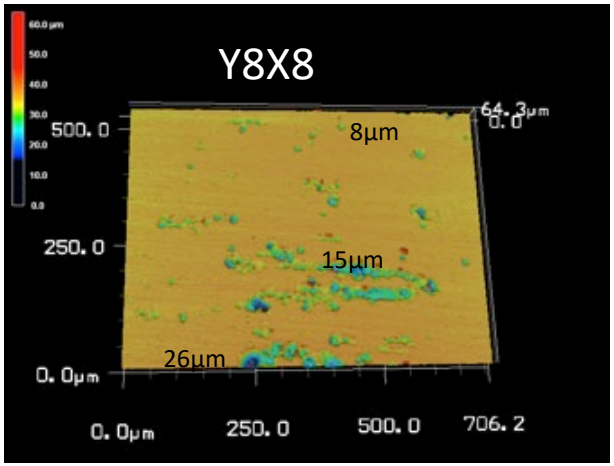
- Pitting on the SAVY piece is spread out
- Pitting on the Hagan piece is localized close to the weld and 2.5 mm below the weld

LCM Analyses on SAVY Specimen



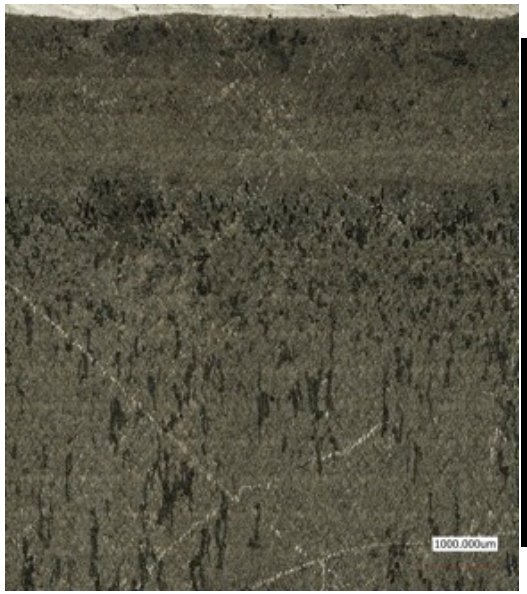


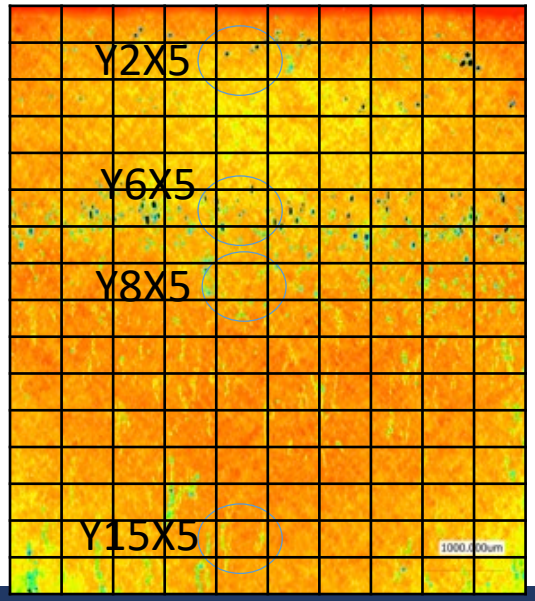


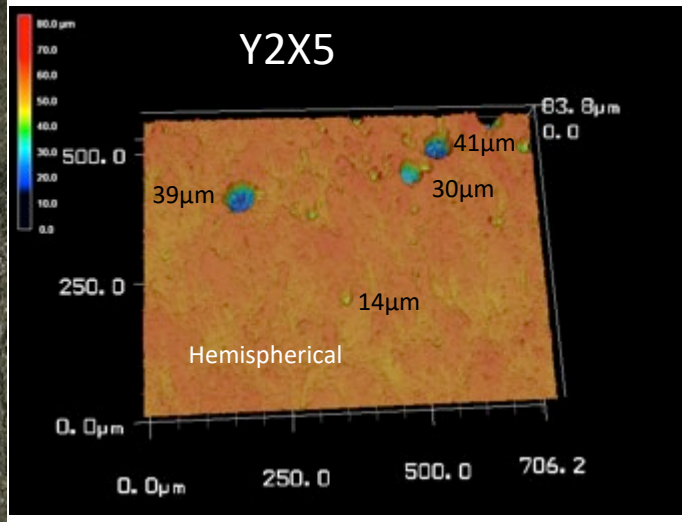


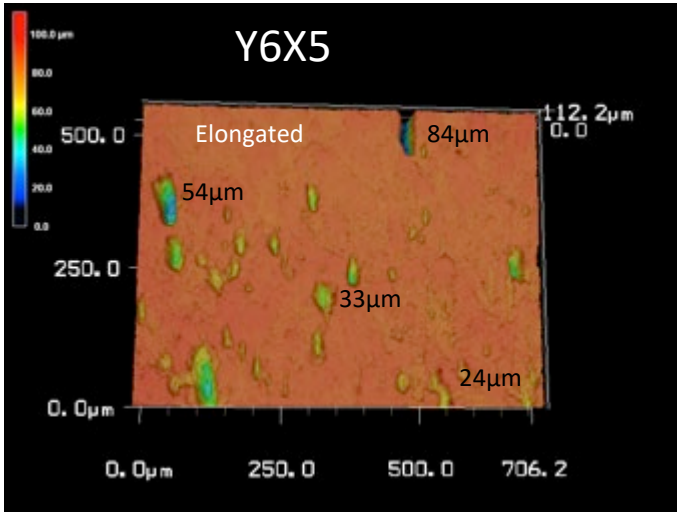
- Pitting is spread out with depths from 8 to 42 µm.

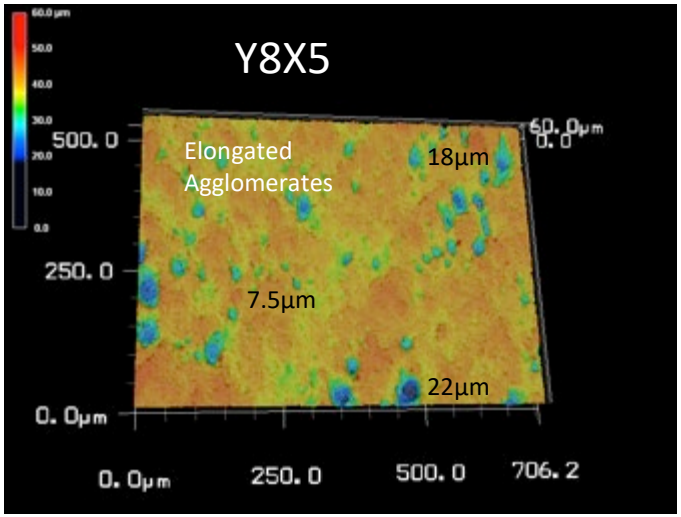
LCM Analyses on Hagan Specimen

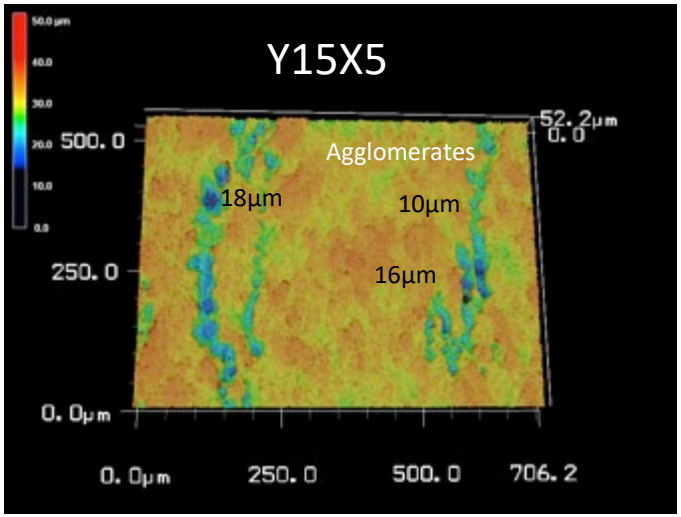








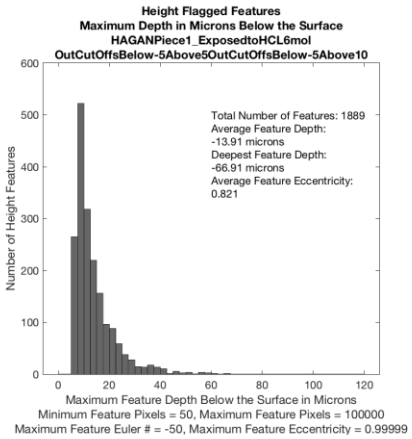


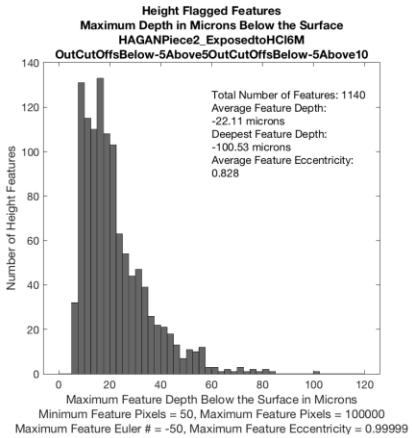


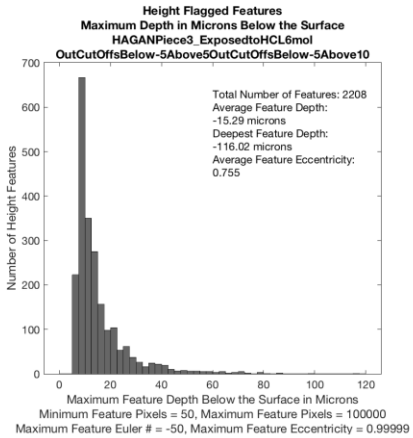
- Pit depths range from 7.5 to 54 µm. Pits close to the weld are round and elongated when away from the weld.

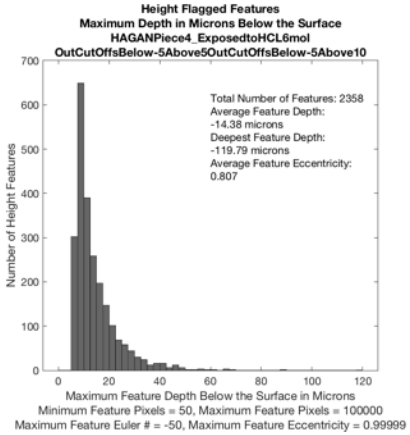
Hagan

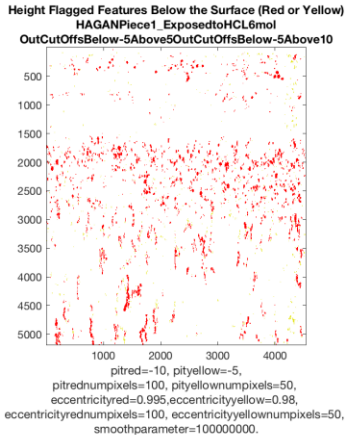
-10/-5

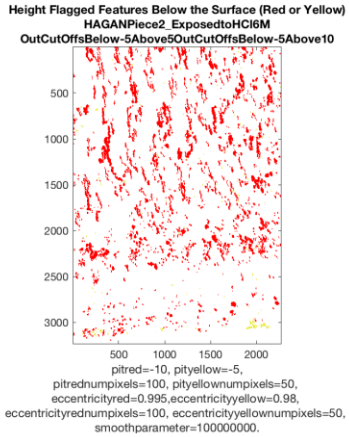

Height Flagged Features
Maximum Depth in Microns Below the Surface
HAGANPiece1_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 1889
Average Feature Depth:
-13.91 microns
Deepest Feature Depth:
-66.91 microns
Average Feature Eccentricity:
0.821
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99999

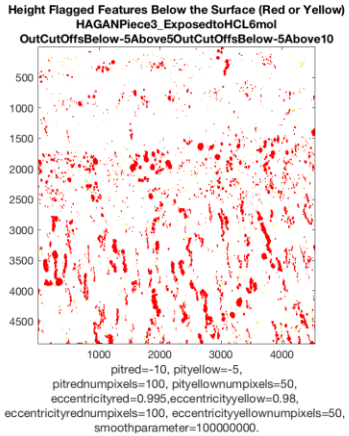

Height Flagged Features
Maximum Depth in Microns Below the Surface
HAGANPiece2_ExposedtoHCl6M
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 1140
Average Feature Depth:
-22.11 microns
Deepest Feature Depth:
-100.53 microns
Average Feature Eccentricity:
0.828
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99999


Height Flagged Features
Maximum Depth in Microns Below the Surface
HAGANPiece3_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 2208
Average Feature Depth:
-15.29 microns
Deepest Feature Depth:
-116.02 microns
Average Feature Eccentricity:
0.755
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99999


Height Flagged Features
Maximum Depth in Microns Below the Surface
HAGANPiece4_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 2358
Average Feature Depth:
-14.38 microns
Deepest Feature Depth:
-119.79 microns
Average Feature Eccentricity:
0.807
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99999


Height Flagged Features Below the Surface (Red or Yellow)
HAGANPiece1_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
smoothparameter=100000000.


Height Flagged Features Below the Surface (Red or Yellow)
HAGANPiece2_ExposedtoHCl6M
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
smoothparameter=100000000.


Height Flagged Features Below the Surface (Red or Yellow)
HAGANPiece3_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
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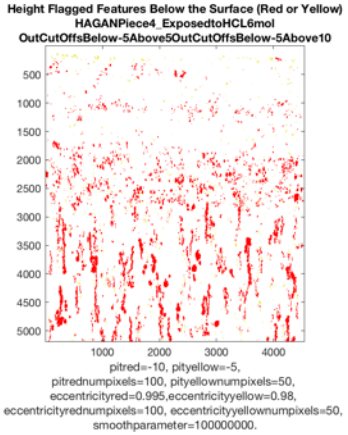
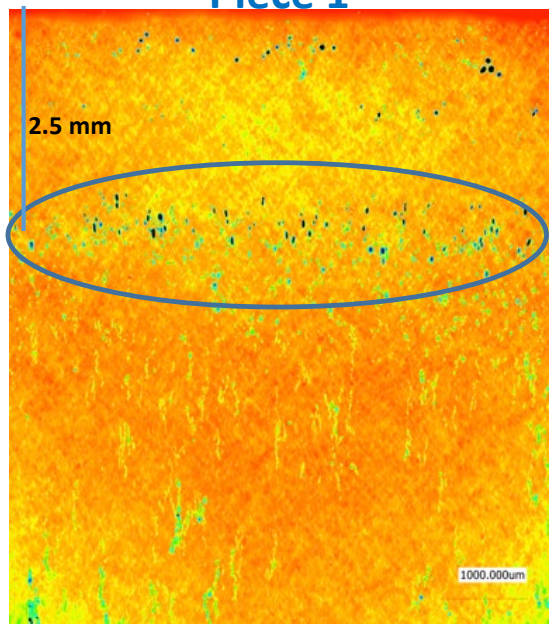
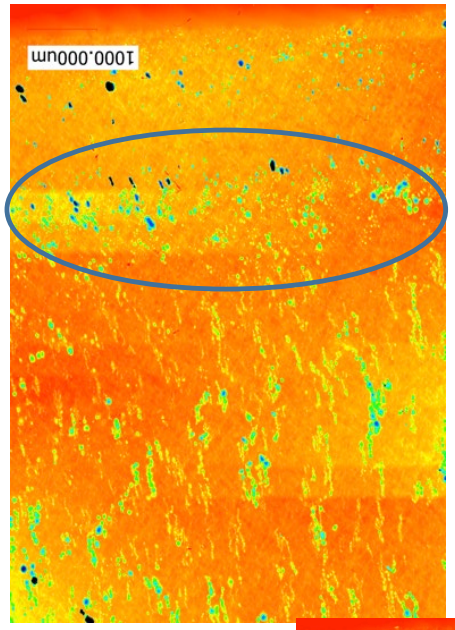

Height Flagged Features Below the Surface (Red or Yellow)
HAGANPiece4_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
smoothparameter=100000000.

LCM Analyses on Hagan Specimens

Piece 1

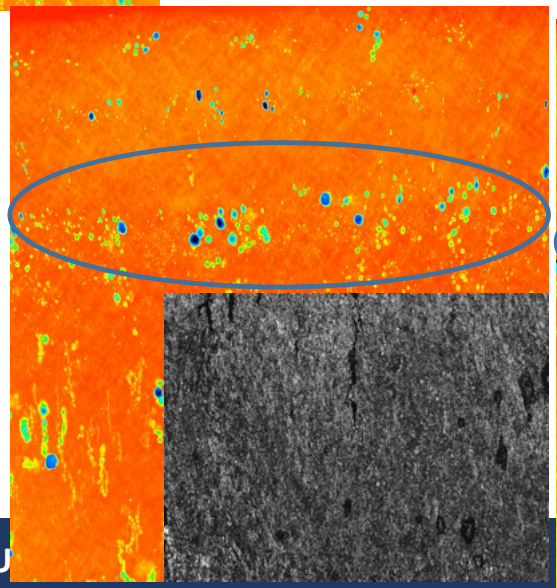


Piece 2



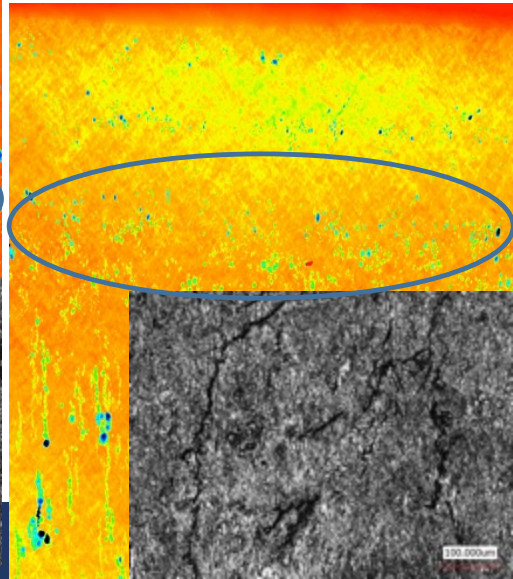
- Lots of pits
- Agglomeration of pits about 2.5mm from weld

Piece 3



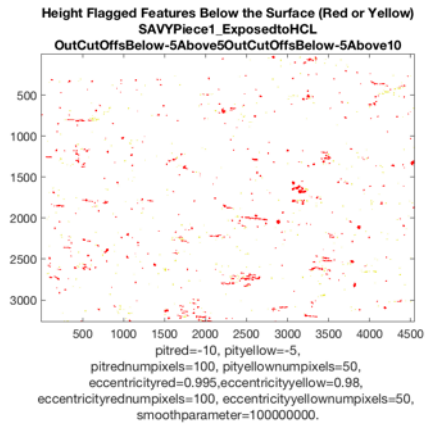
- Lots of pits
- Agglomeration of pits about 2.5mm from weld
- Cracks

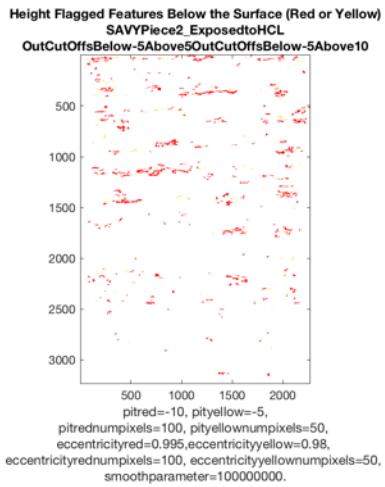
Piece 4

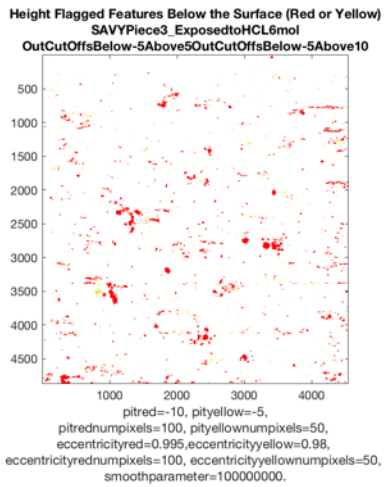


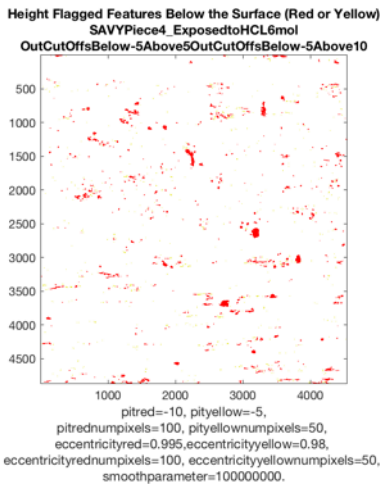
SAVY

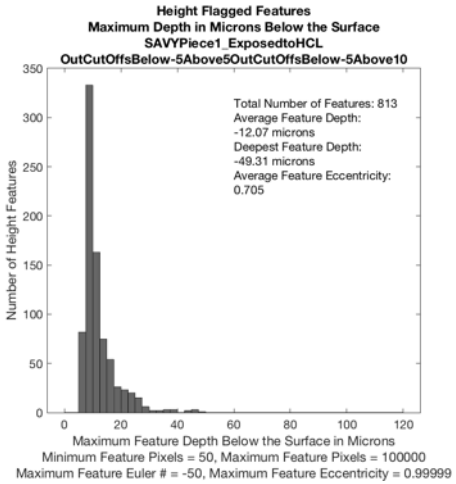
-10/-5

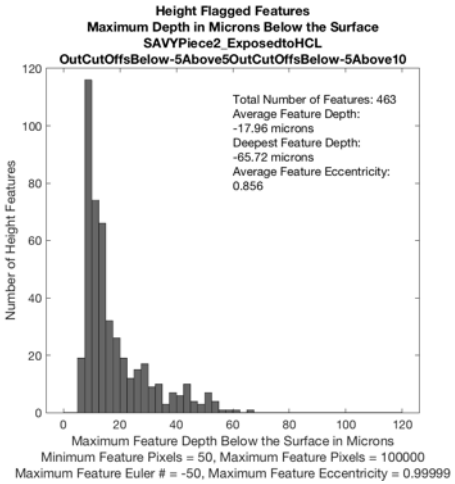

Height Flagged Features Below the Surface (Red or Yellow)
SAVYPiece1_ExposedtoHCL
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
smoothparameter=100000000.

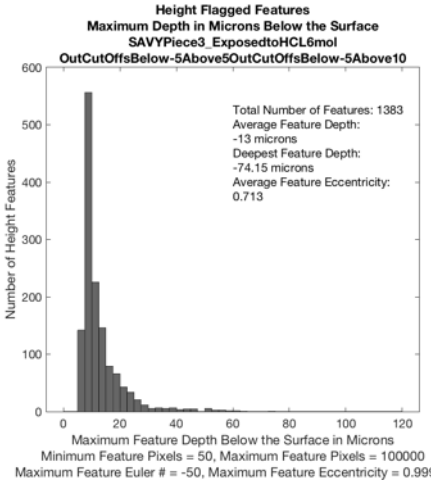

Height Flagged Features Below the Surface (Red or Yellow)
SAVYPiece2_ExposedtoHCL
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
smoothparameter=100000000.

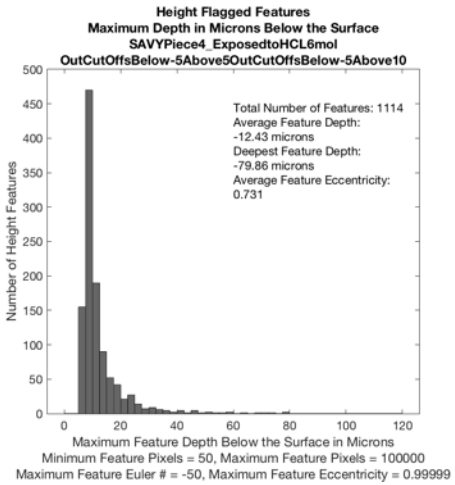

Height Flagged Features Below the Surface (Red or Yellow)
SAVYPiece3_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
smoothparameter=100000000.


Height Flagged Features Below the Surface (Red or Yellow)
SAVYPiece4_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
pitred=-10, pityellow=-5,
pitrednumpixels=100, pityellownumpixels=50,
eccentricityred=0.995,eccentricityyellow=0.98,
eccentricityrednumpixels=100, eccentricityyellownumpixels=50,
smoothparameter=100000000.

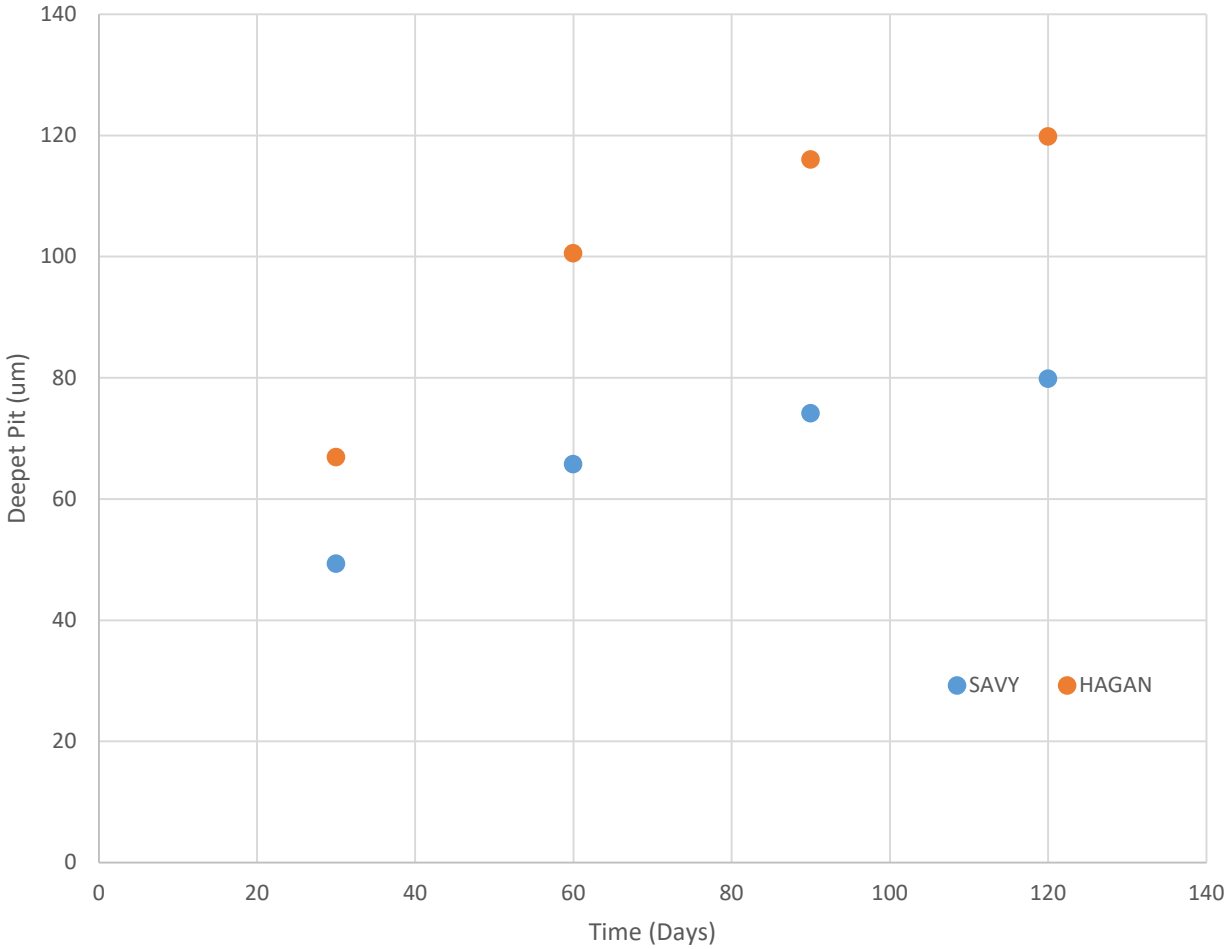

Height Flagged Features
Maximum Depth in Microns Below the Surface
SAVYPiece1_ExposedtoHCL
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 813
Average Feature Depth:
-12.07 microns
Deepest Feature Depth:
-49.31 microns
Average Feature Eccentricity:
0.705
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99999


Height Flagged Features
Maximum Depth in Microns Below the Surface
SAVYPiece2_ExposedtoHCL
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 463
Average Feature Depth:
-17.96 microns
Deepest Feature Depth:
-65.72 microns
Average Feature Eccentricity:
0.856
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99999

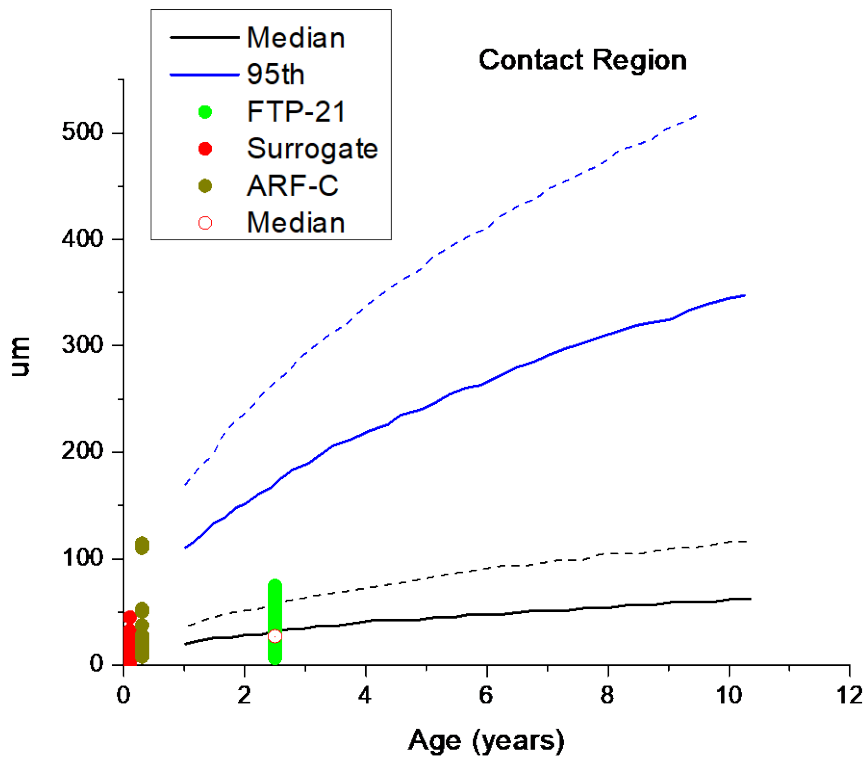

Height Flagged Features
Maximum Depth in Microns Below the Surface
SAVYPiece3_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 1383
Average Feature Depth:
-13 microns
Deepest Feature Depth:
-74.15 microns
Average Feature Eccentricity:
0.713
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99

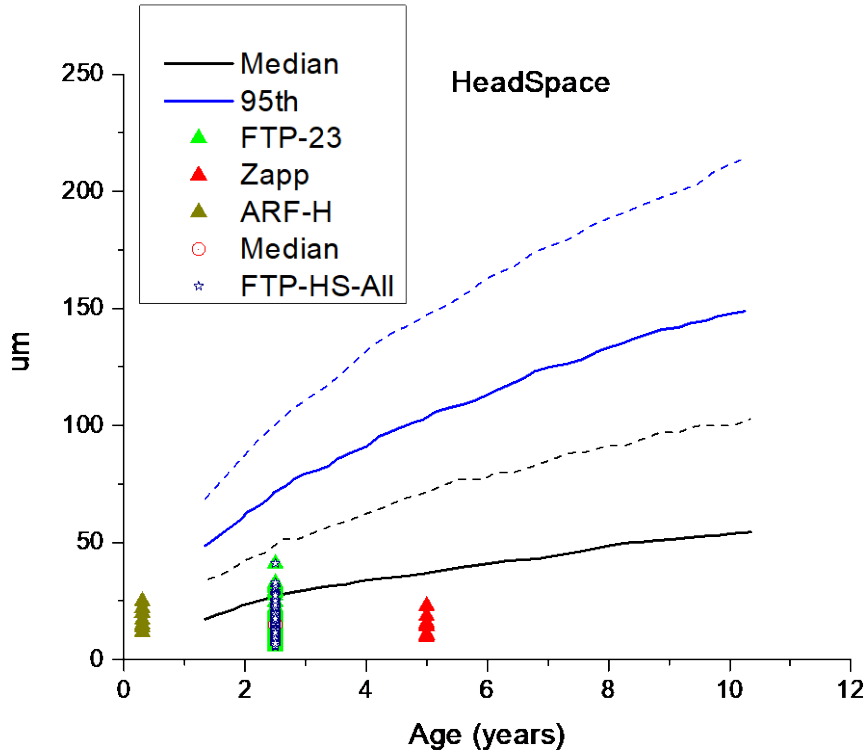

Height Flagged Features
Maximum Depth in Microns Below the Surface
SAVYPiece4_ExposedtoHCL6mol
OutCutOffsBelow-5Above5OutCutOffsBelow-5Above10
Total Number of Features: 1114
Average Feature Depth:
-12.43 microns
Deepest Feature Depth:
-79.86 microns
Average Feature Eccentricity:
0.731
Number of Height Features
Maximum Feature Depth Below the Surface in Microns
Minimum Feature Pixels = 50, Maximum Feature Pixels = 100000
Maximum Feature Euler # = -50, Maximum Feature Eccentricity = 0.99999

Pit Growth


140
120
100
80
60
40
20
0
Deepet Pit (um)
0
20
40
60
80
100
120
140
Time (Days)
SAVY
HAGAN

Pit growth model



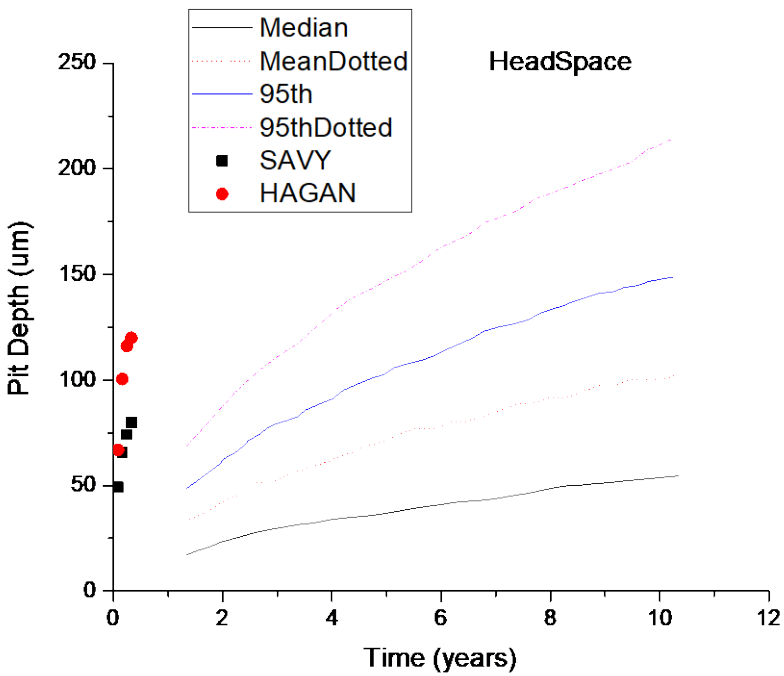


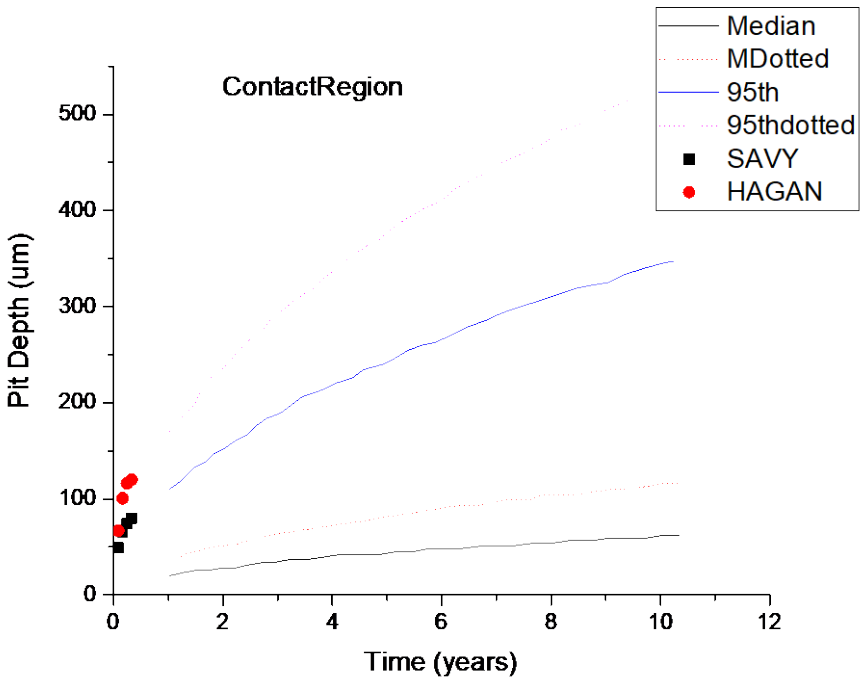
Model introduced by Elizabeth

Kelly, E. J.; Graves, T. L.; Veirs, D. K.; Duque, J. G.; Berg, J. M.; Worl, L. A.; Mickalonis, J. I. (2014-10-17). "A Statistical Framework for Comparison of Predictions of Corrosion Pit Depth Growth Based on Multiple Data Sets Collected Under Different Experimental and Environmental Conditions," Los Alamos National Laboratory, LA-UR-14-28129.

Kelly, E. J.; Graves, T. L.; Veirs, D. K.; Berg, J. M.; Duque, J. G.; Worl, L. A.; Mickalonis, J. I. (2013-07-23). "A General Statistical Model for Corrosion Pit Depth Analysis for Threshold Data," Los Alamos National Laboratory, LA-UR-13-25127

Pit growth model: SAVY-HAGAN





Summary

SAVY:

- Corrosion on metal specimens increases with increasing exposure time. No cracking is observed on these samples.
- Corrosion density on the container body and lid also increases with exposure time.

Hagan:

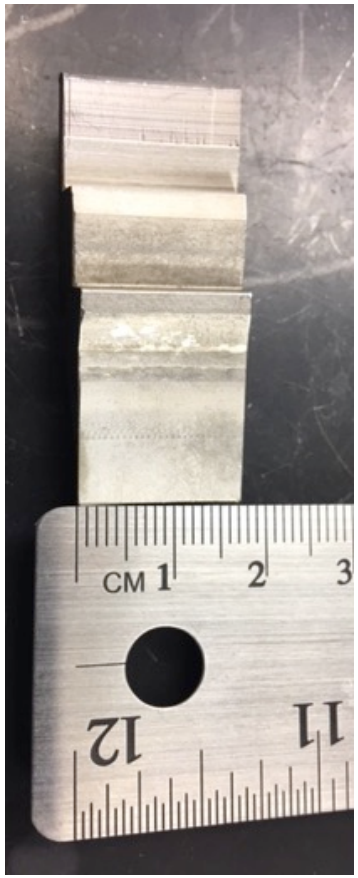
- Corrosion on metal specimens increases with increasing exposure time. Cracking is observed on the 4th sample.
- Corrosion density on the container body and lid also increases with exposure time.

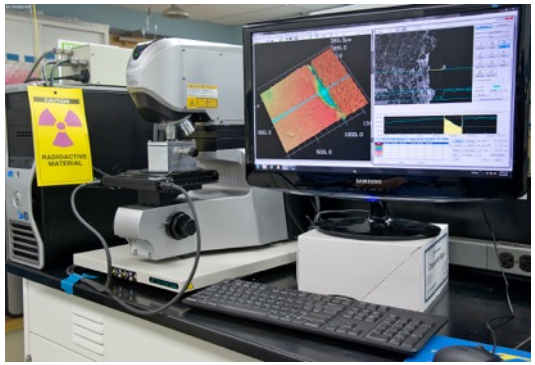
Path forward

- Analyses of containers by non-destructive methods?
  - Ultrasonic testing
  - Eddy current
  - X-ray tomography
- Containers to drop tested to determine if containers with this much corrosion damage resist a drop?
- Analyses of containers by LCM and metallography

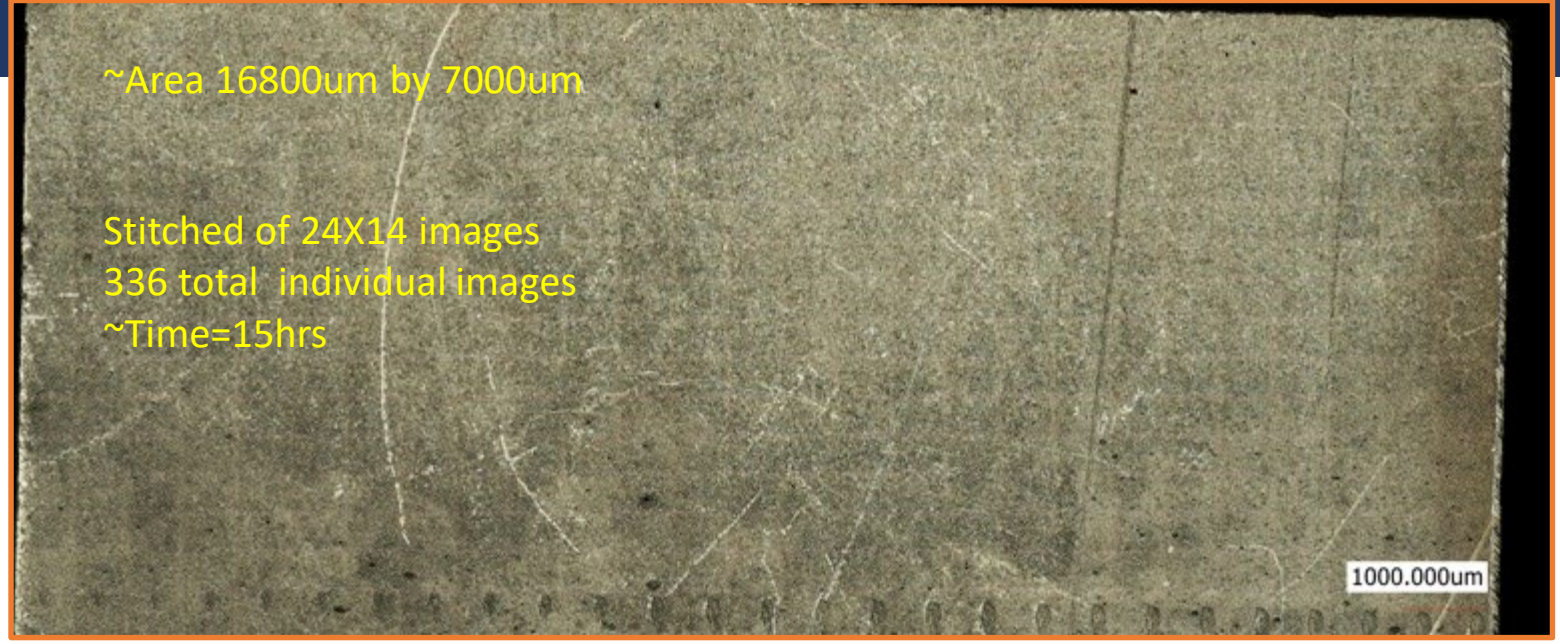
SAVY: SAI

Section from weld region





LCM imaged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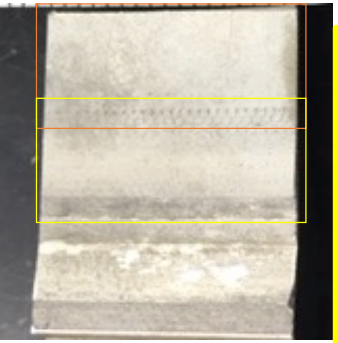
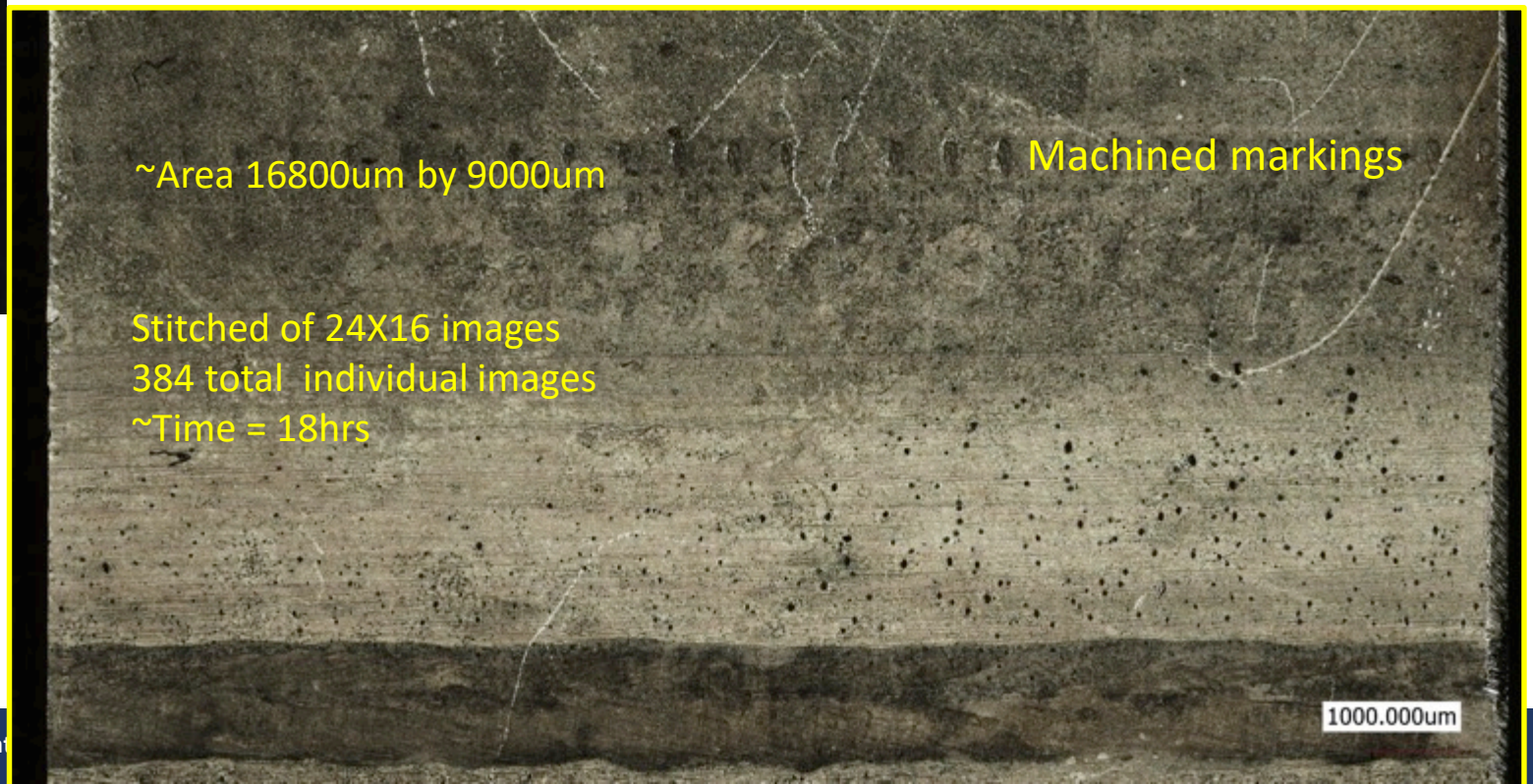
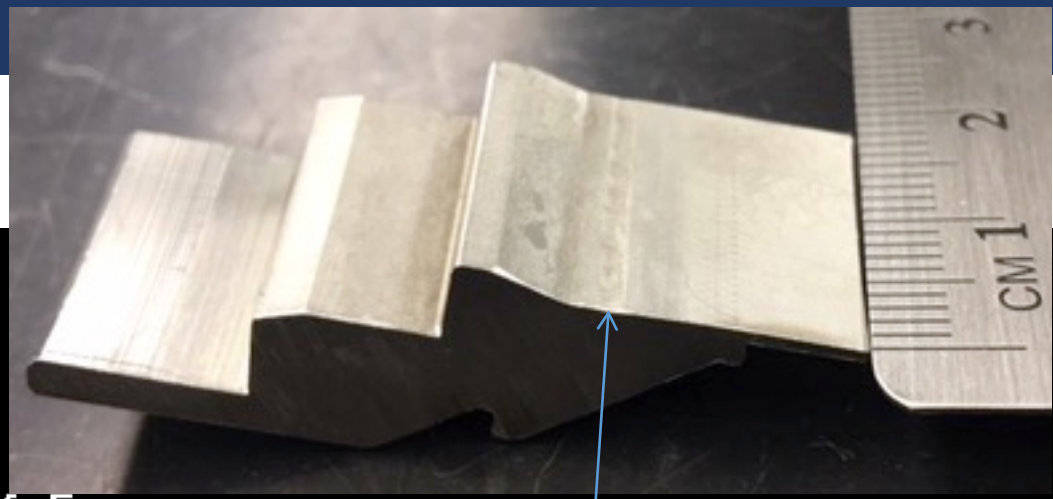
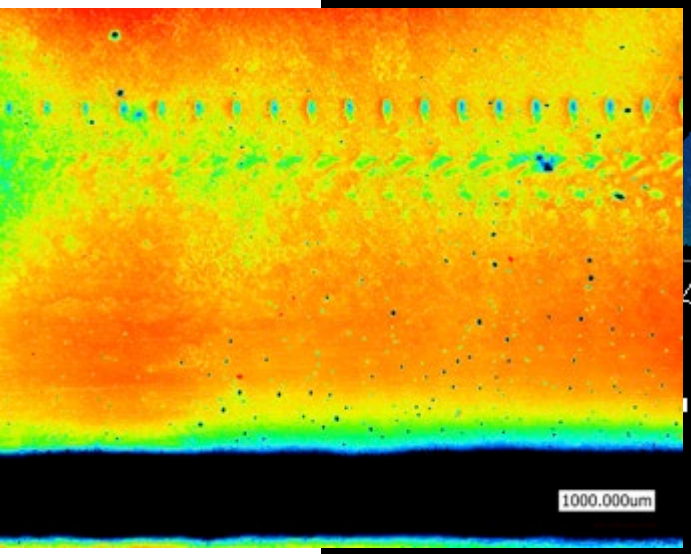
Stitched of 24X14 images
336 total individual images
~Time=15hrs
1000.000um

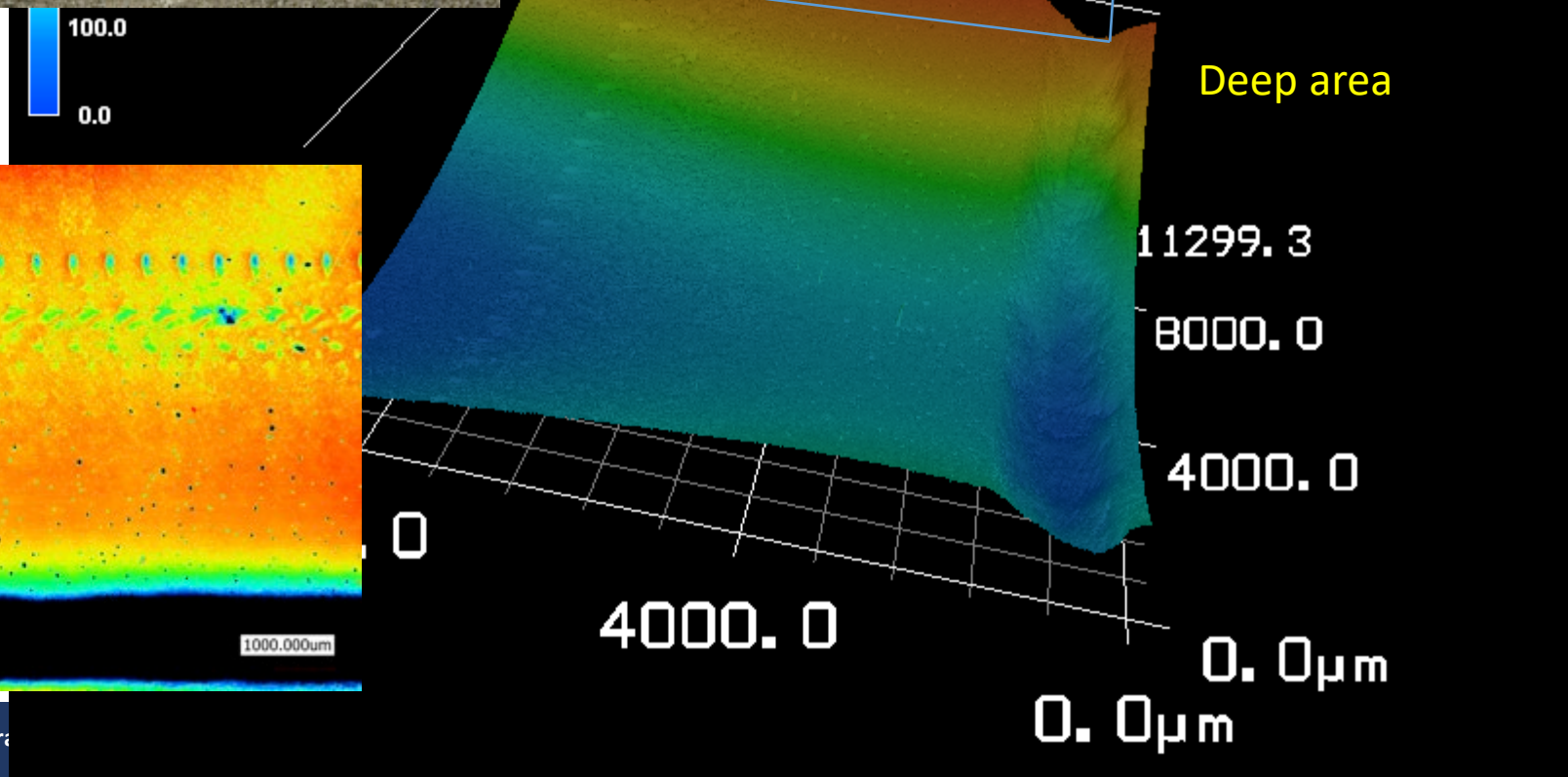


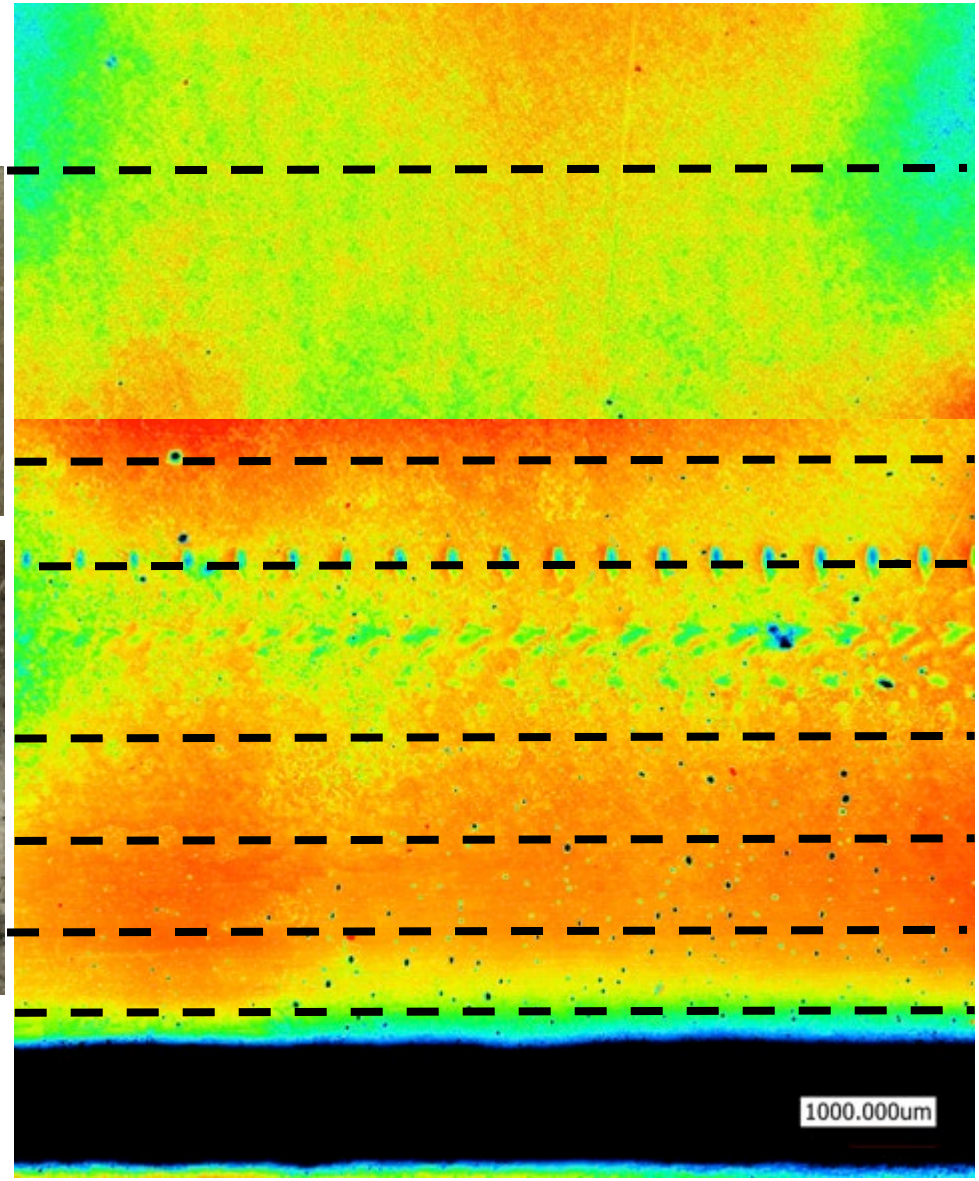

~Area 16800um by 9000um
Machined markings
Stitched of 24X16 images
384 total individual images
~Time = 18hrs
1000.000um

Trimmed
1000.000um


CM 1
2
3


1000.000um


576.5µm
100.0
0.0
Deep area
11299.3
8000.0
4000.0
0
4000.0
0.0µm
0.0µm

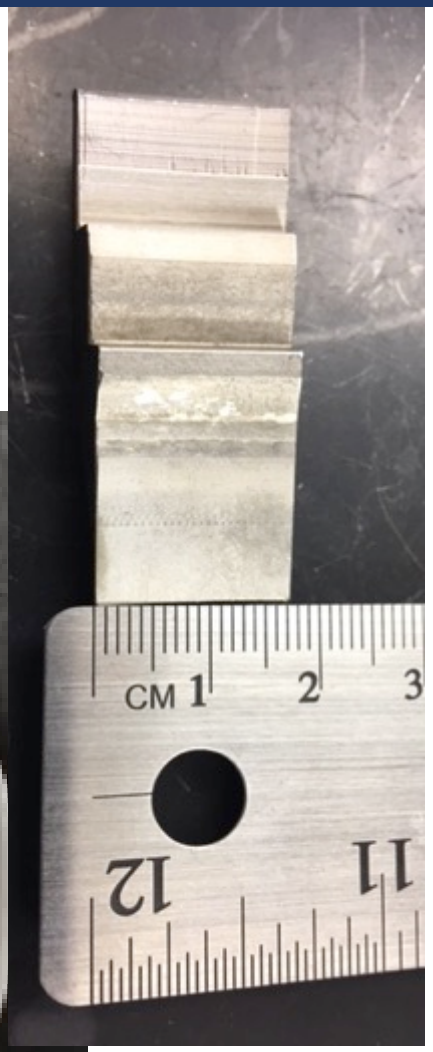


Increasing distribution of pits

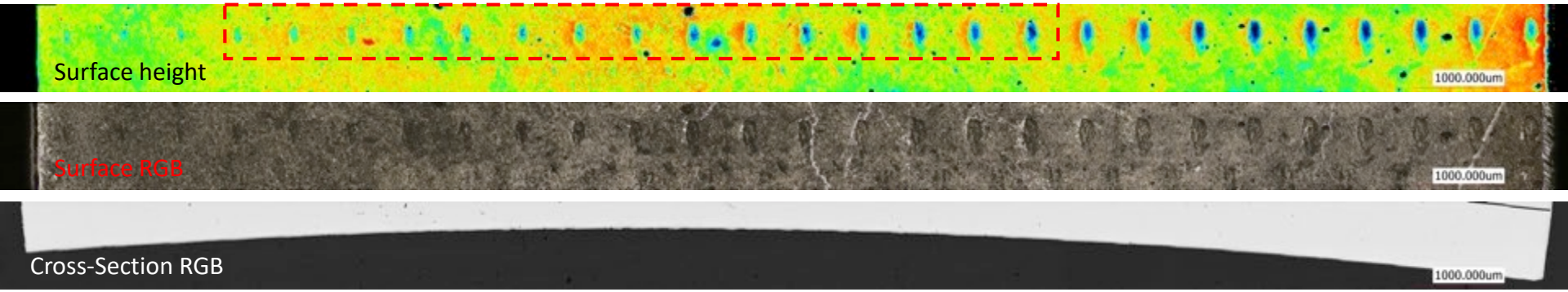
Idea will be to do cross-sections away from weld and multiple below machine markings.
Dotted lines represent approximated cross-sections

Cross-Section

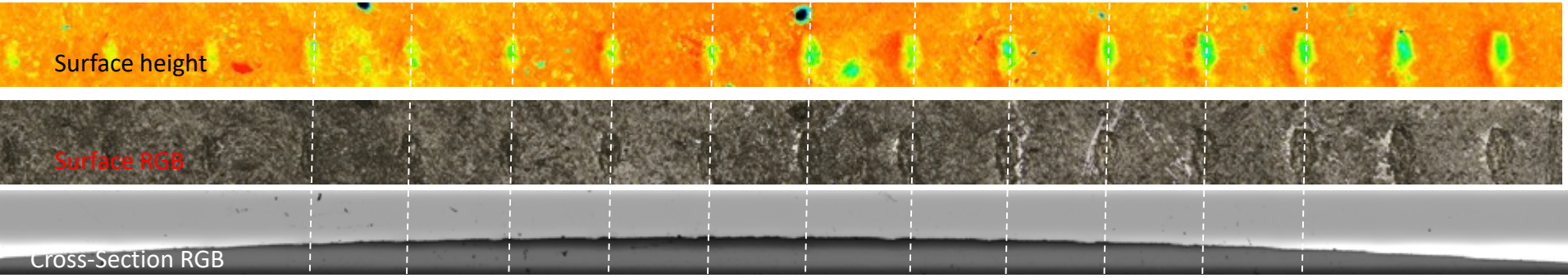
Thanks to MaryAnn Hill



Cross-Section 4
~6.5mm from top

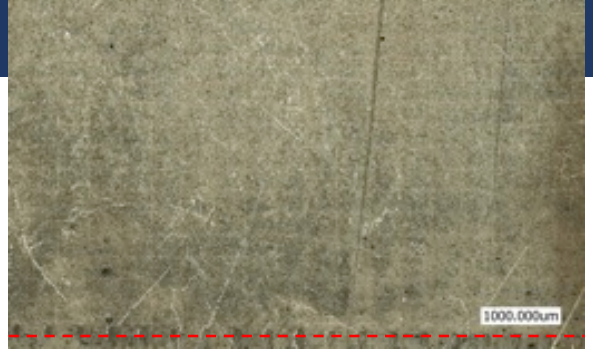


Not sure if I am at the top or bottom of these machine marks

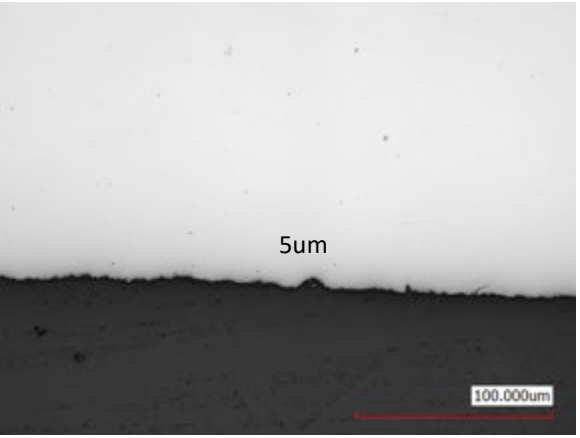


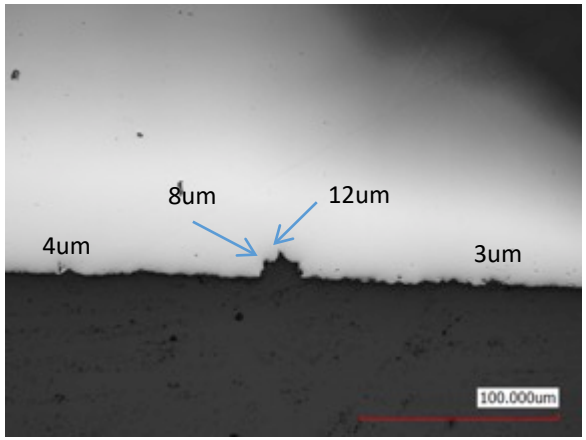
- Good correlation between images from surface and cross-section (white lines)

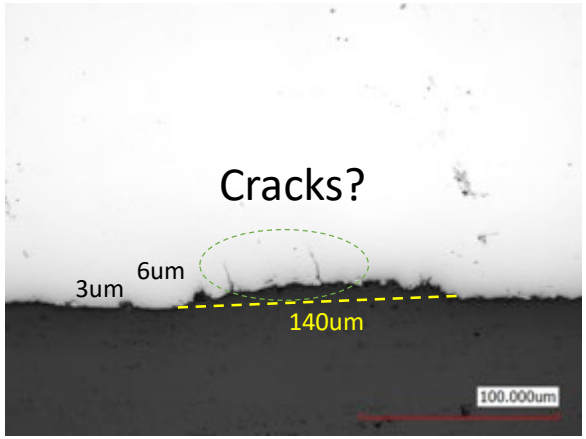
Cross-Section 4
~6.5mm from top



6.5mm







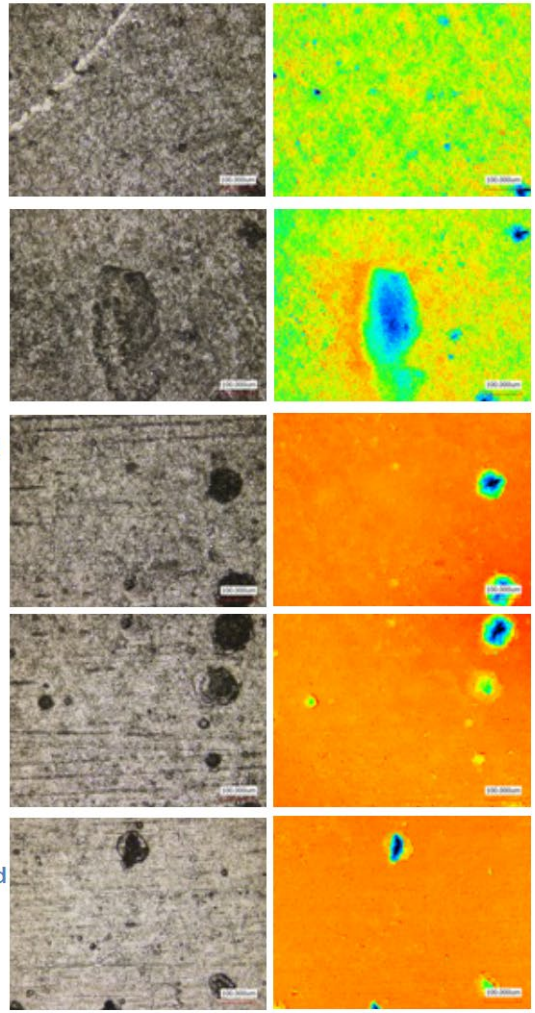
Overall Observations

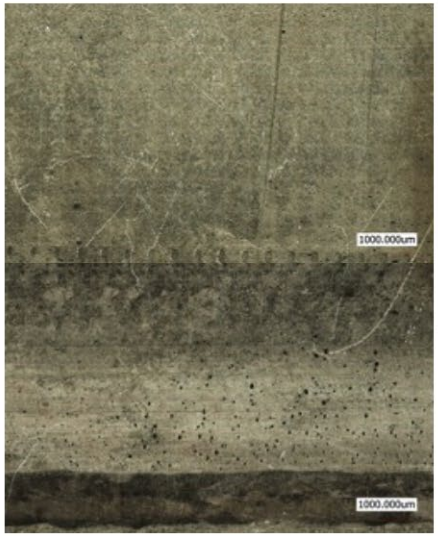
Surface

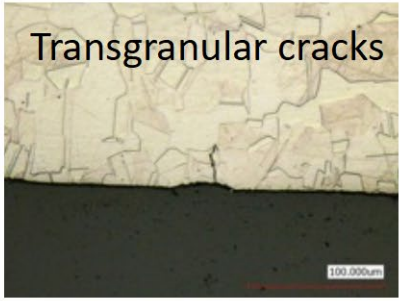
Mild surface damage

Rounded pits

Elongated pits





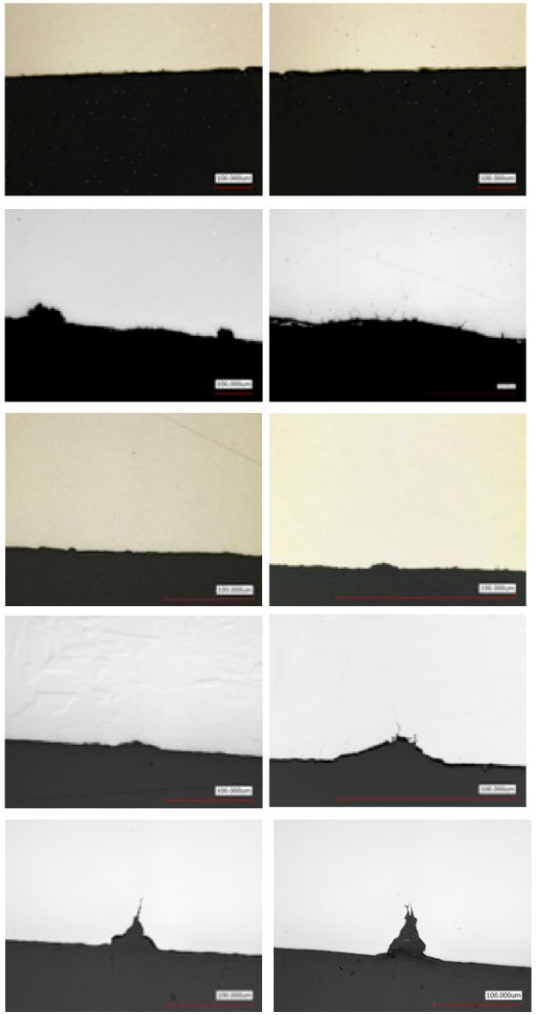


Cross-Section

Mild surface damage

Rounded pits

Elongated pits with cracks



SAVY Inventory Planning and Procurement

NMP Program Review

Zack Smith (Industrial and Systems Engineering, SPE-2)

8/20/2019

Operated by Los Alamos National Security, LLC for the U.S. Department of Energy's NNSA

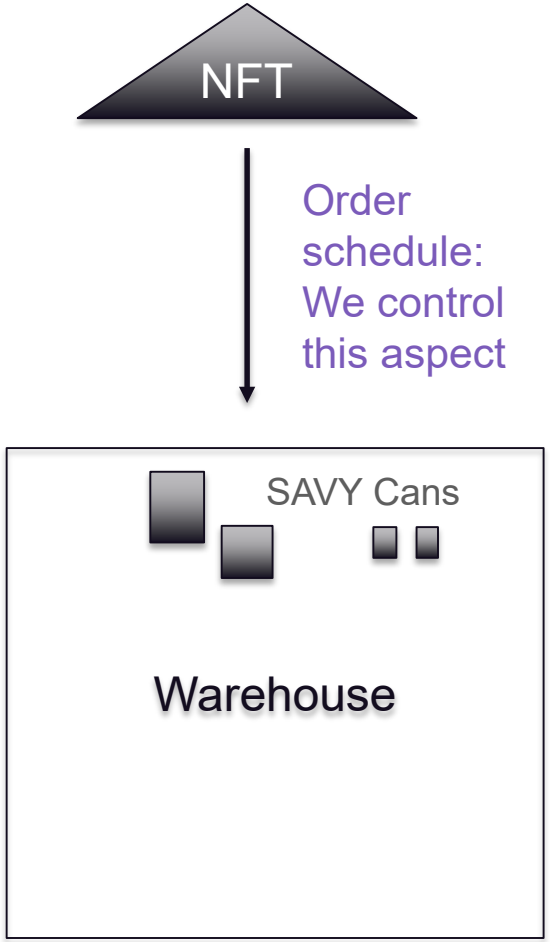
Presentation Outline

- Background/SAVY Procurement Problem Description
- Historical SAVY Purchase Data
- Subjective SAVY Demand Forecasting
- Future SAVY Order Planning

Background on SAVY Procurement

- **The container team is responsible for procuring SAVYs for LANL's manufacturing and science mission, as well as for the rest of the DOE complex.**
- **Due to unforeseen changes in SAVY usage, we have had container shortages.**
- **The goal of this project: develop inventory management tools to adequately and efficiently supply SAVY containers.**
- **Project Successes:**
  - Established continuous monitoring of SAVY inventory levels.
  - Established continuous monitoring of SAVY demand data.
  - Obtained subjective SAVY demand forecasts from the biggest users.
  - Developed simple SAVY order planning tools.

An Illustrative Picture of the Decision Problem



We decide:
What type of cans to order?
When?
How many?

Key Input: To make good ordering decisions, we have to project future programmatic demand

"I want K 1Qt SAVYs"

Demand from programs

On the other hand, there is an opportunity cost for holding massive inventories, or manufacturer constraints may preclude us from building large reserves.

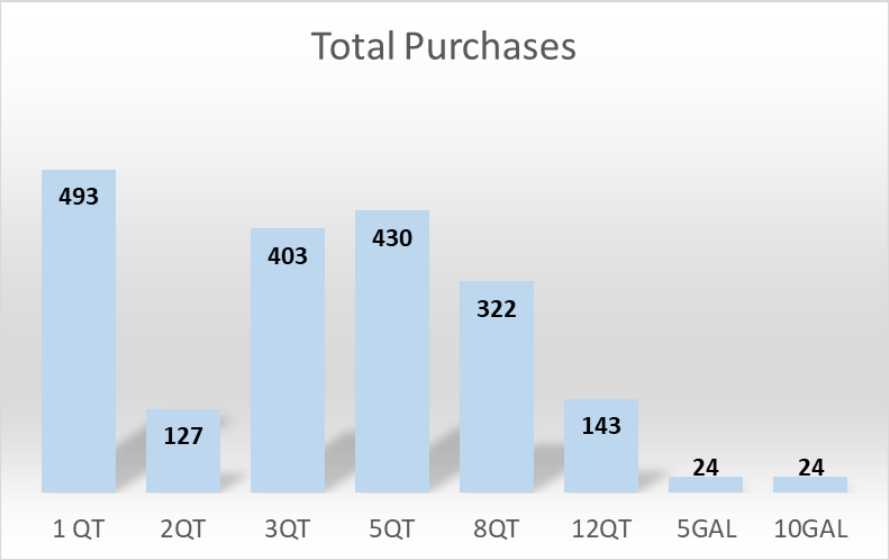
In the LANL setting, not having adequate supply of containers can delay or halt production with massive associated costs

What are some of the unique challenges?

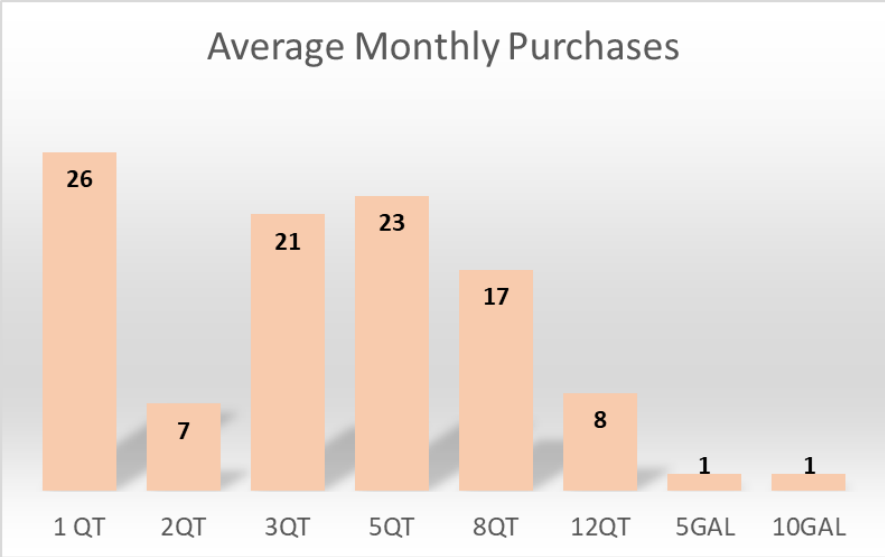
- **Random, time-dependent demand for SAVYs.**
  - Increase in manufacturing activities.
  - Changes in usage and recycling protocols.
- **Demand for one SAVY type depends on the quantity available of other containers types.**
  - In particular, larger containers are potential substitutes for smaller containers.
  - This means that demand is decision-dependent; our ordering schedule impacts how inventory is used.
- **Long ordering lead times.**
- **Segmented inventory pools.**

Historical SAVY Purchase Data

- **Data source: Oracle Warehouse Transactions Records (Jan. 2018 – Jul. 2019).**



Historically, 1QT and 5QTs have been the most purchased container types.

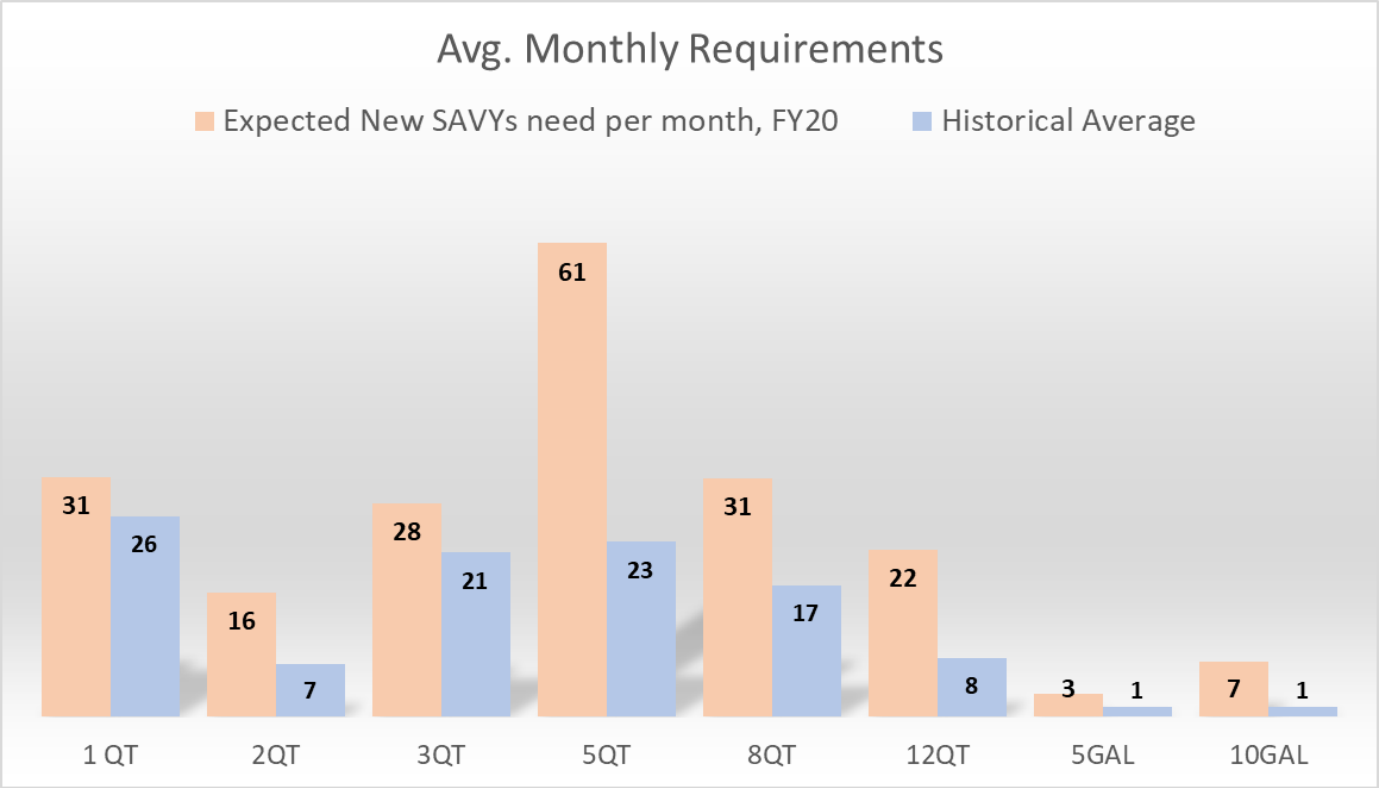


Originally, we modeled future demand based on historical data.

Concern from SMEs that future SAVY needs would not resemble past SAVY needs.

So, we obtained subjective forecasts for future new SAVY needs from the groups that use SAVYs the most

- **Data source: Subjective Estimates for new SAVYs required in FY20 (we also obtained estimates for FY19 and FY21).**



We need ***more*** new SAVYs then relative to historical purchasing.

The ***distribution*** of new SAVYs required differs from historical purchases.

How do we turn our demand forecast into a schedule for order quantities and receipt dates?

Data

Demand forecast or model

Holding costs, shortage costs, purchase costs

Assumptions

Demand substitution between sizes

Model

Constraints

Budget constraints

Manufacturer constraints

Planned Receipt Schedule

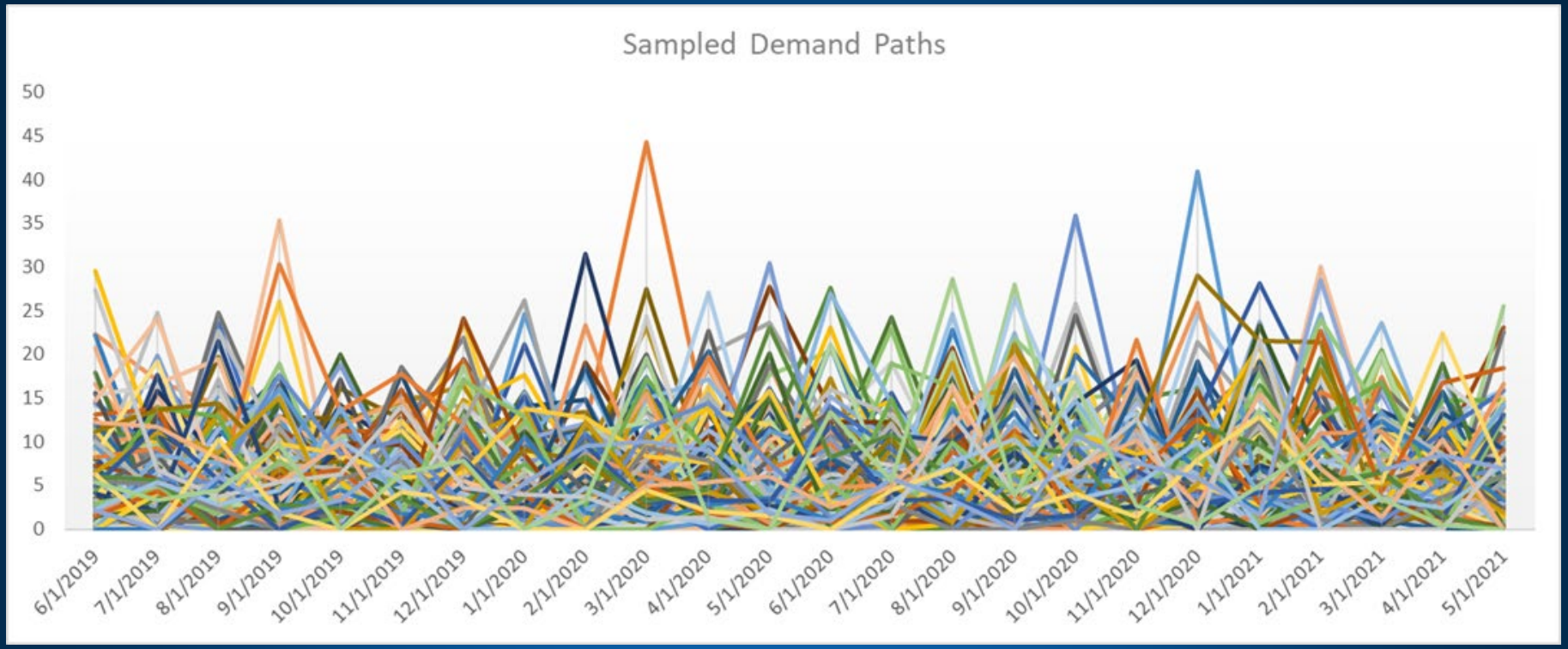
SAVY	Date
1QT	50
5QT	150

Current model: Simple, tractable, explainable

- **Deterministic Demand Schedule from Subjective Forecasts.**
  - However, the demand path is conservative; high probability of carrying extra stock.
  - Advantage: simple to explain how the purchases correspond with the subjective forecasts.
  - Recognition that holding costs/ extra supply is less problematic than running out of SAVYs/ halting production.
- **Limited Recourse.**
  - We do not place orders reactively after observing demand due to the onerous contractual process for new orders.
- **Manufacturing/Budget Constraints.**
  - Assume none
  - The problem reduces to satisfying all demand at the lowest cost

A note on demand modeling

- **Modeling the demand process itself can be complex. However, many inventory models perform poorly when real-world randomness is not incorporated.**
  - One could develop a (simulation) model of the stochastic demand process.
  - Random inter-arrival times to warehouse, random order quantities, probabilistic container substitution.
  - Time inhomogeneity in demand due to changing usage patterns.
  - Problem: Hard to plan against.
- **Alternative: ex-post sensitivity analysis.**
  - Stress test your purchase schedule against a random demand model.



Thank You. Questions?

Acknowledgements: Jude Oka, Tim Stone, Jim Rubin, Paul Smith, Sonia Parsons.
Thank you for your help with this effort!

Overview of SAVY 4000 Pressurization Simulations

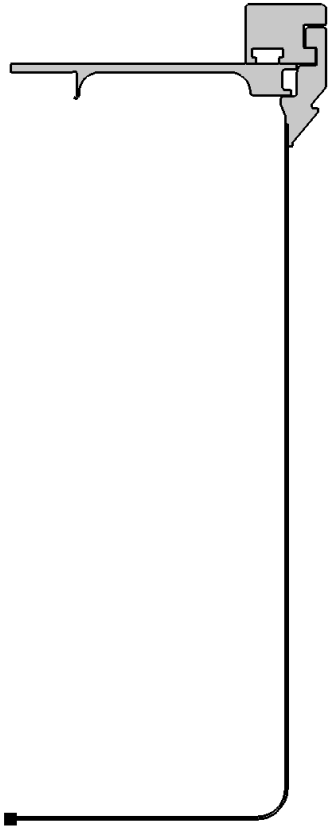
Jamie Kimberley
Department of Mechanical Engineering
Energetic Materials Research and Testing Center
New Mexico Tech

Overview of Project

- NMT is investigating the response of SAVY containers subjected to internal pressurization which may result from internal gas generation and a blocked filter port
- Finite element simulations have been conducted to evaluate the internal pressurization rate and magnitude required to cause mechanical failure of the container
- Expected companion experiments:
  - Hydrostatic pressurization to failure
  - Rate-controlled Helium pressurization to evaluate seal leak/diffusion
  - Oxide powder simulant with tracer doping to evaluate particulate leakage
- This presentation focuses on the FE simulations

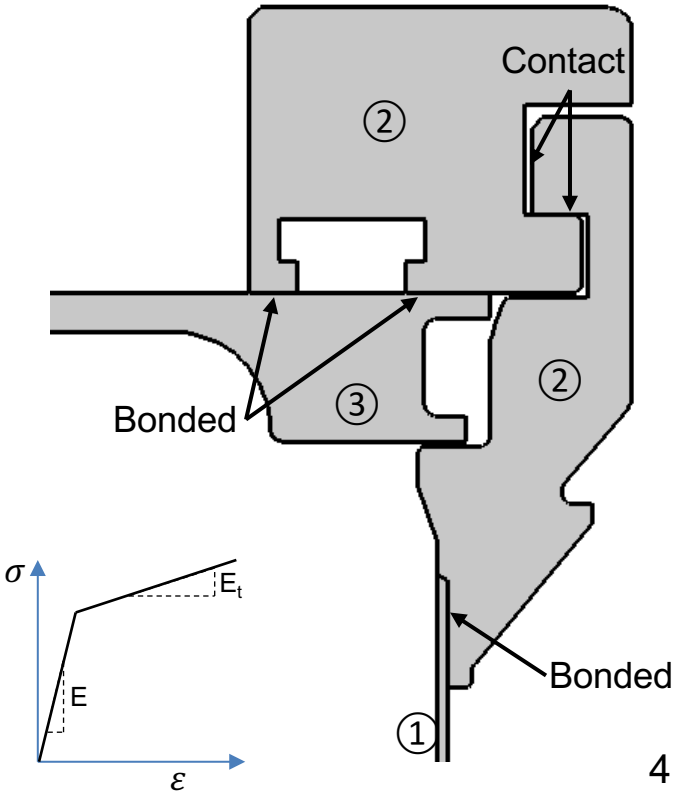
Finite Element Simulation Geometry

- Imported LANL-provided CAD of 5 qt container
- Model a “slice” as axisymmetric
  - Container is not truly axisymmetric but the significant deformation occurs in a region that is axisymmetric
- Small components and features removed
  - O-ring
  - Filter holes
  - Filter pack
  - Handle



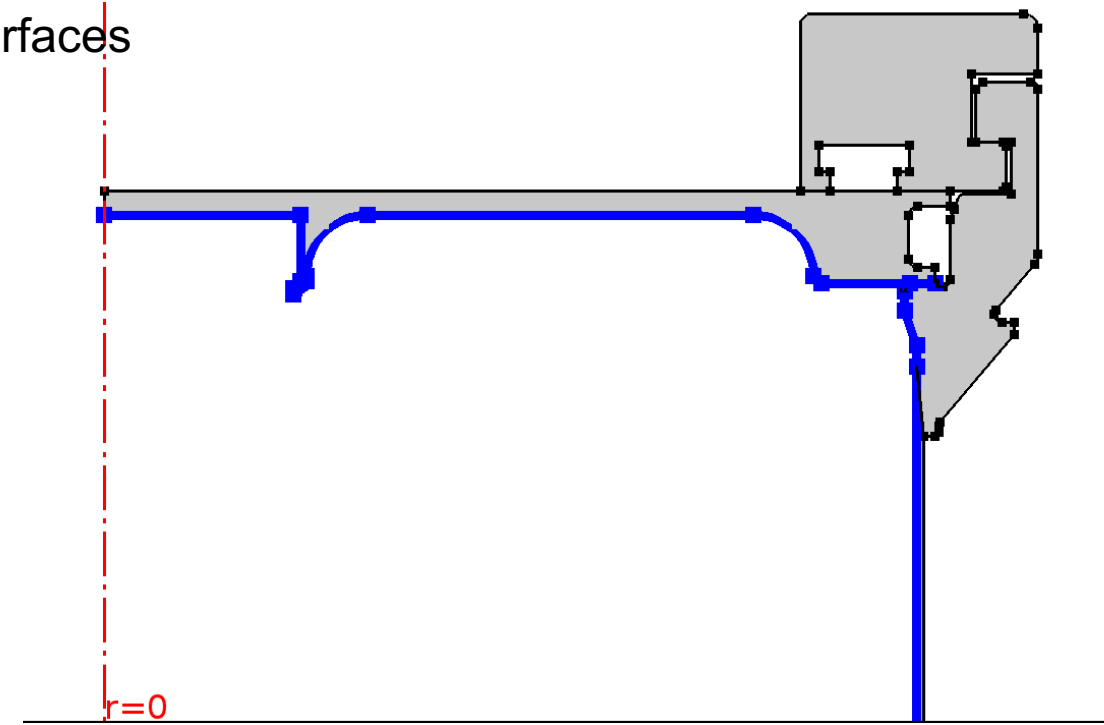
Component Connections and Materials

- Lid and locking ring considered bonded
- Upper and lower can body bonded
- Contact between upper can and locking ring
- All materials 316L SS
  - Young's modulus: 193.8 GPa, 28,000 ksi
  - Poisson's ratio: 0.294
- Isotropic plasticity with linear hardening based on LANL provided material acceptance report
  - ① Body: E_t = 259.5 ksi, ε_{max} = 0.35
  - ② Locking collar: E_t =208.8 ksi, ε_{max} = 0.54
  - ③ Lid: E_t = 208.2 ksi, ε_{max} = 0.41



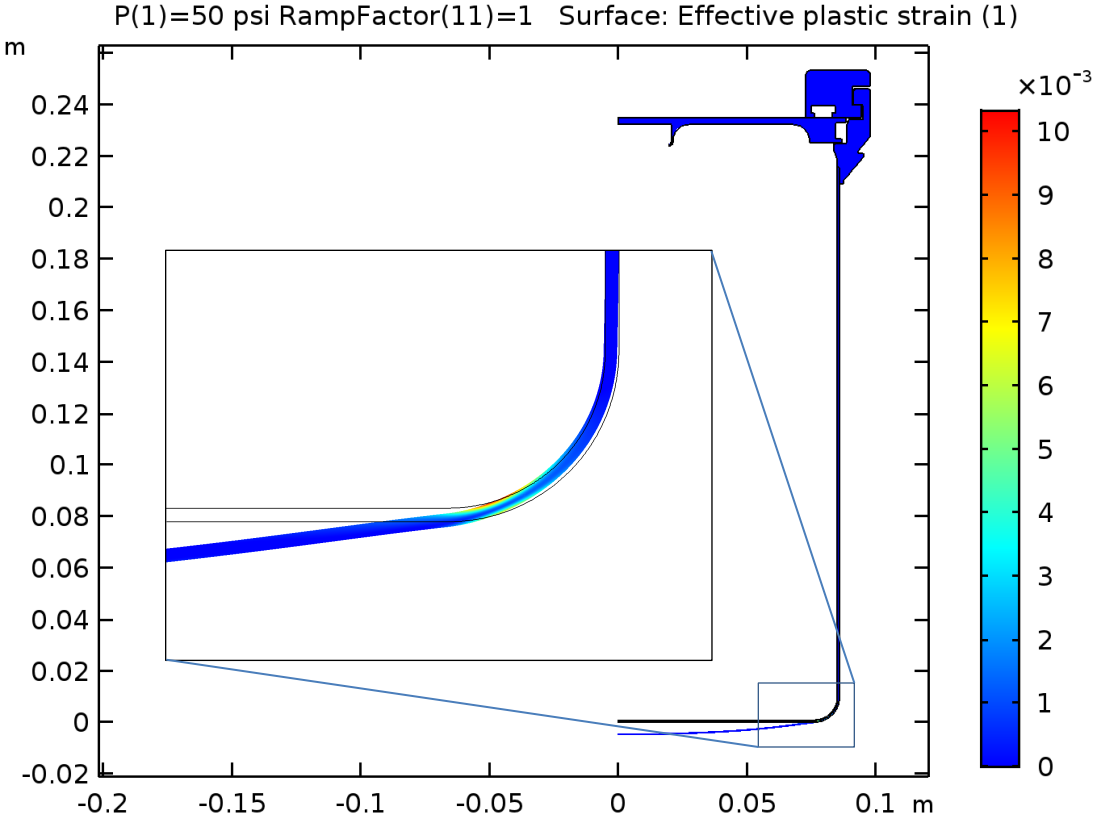
Boundary Conditions

- Pressure applied to internal surfaces
- Values (psi)
  - 50
  - 100-900 in 100 psi steps
  - 950-1050 in 50 psi steps



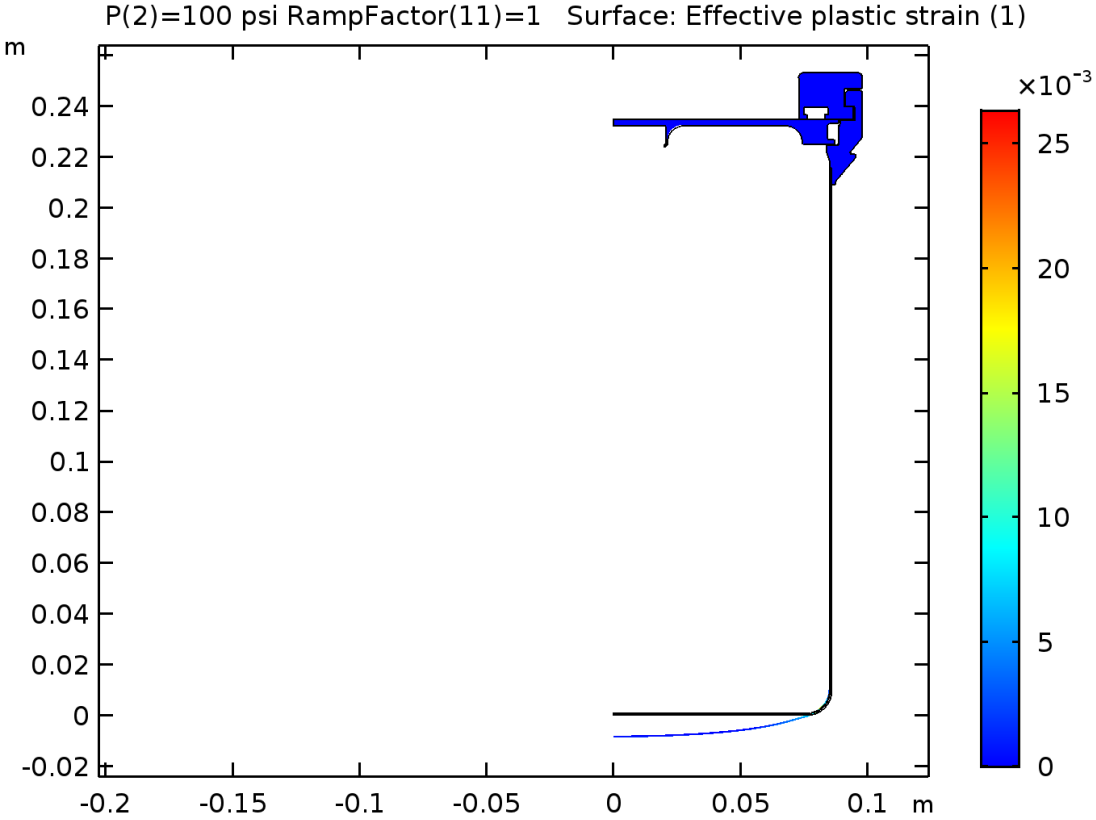
Results: 50 PSI

- Minor yielding in the lower can radius
- Base deflection ~5 mm



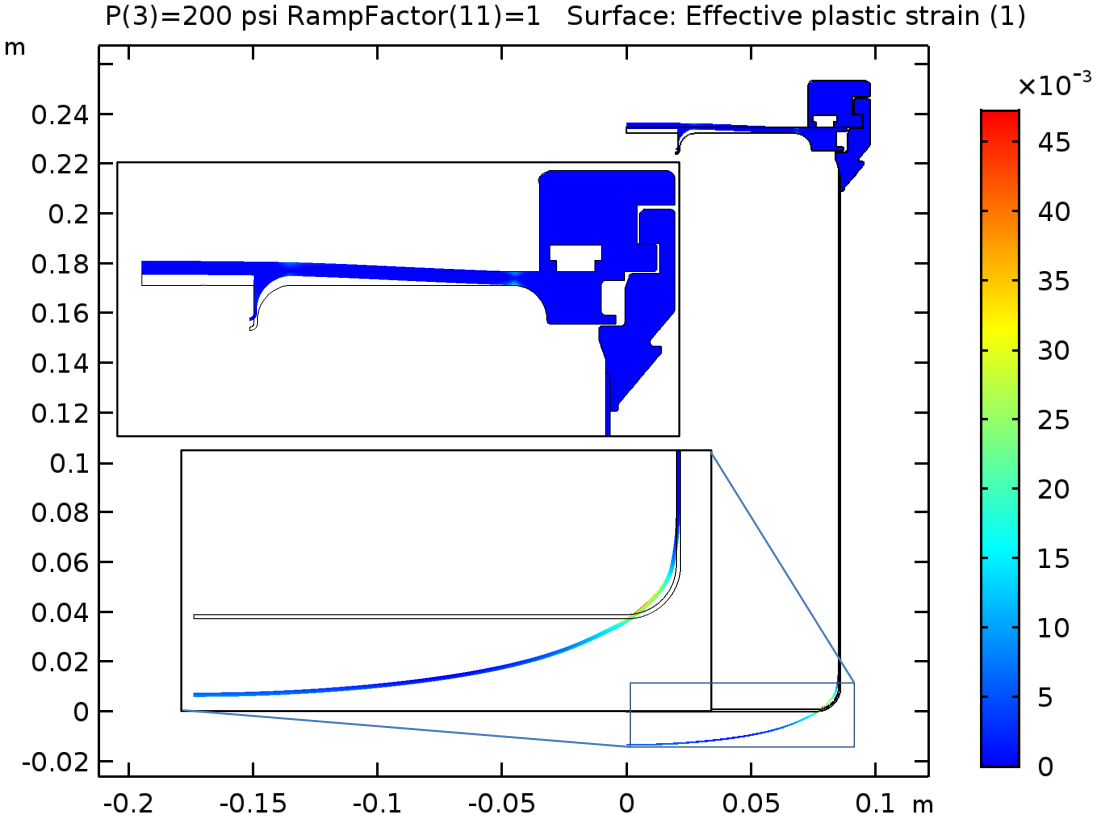
Results: 100 PSI

- Base of container continues to expand and extent of plastic strain spreads
- Max deflection ~9 mm



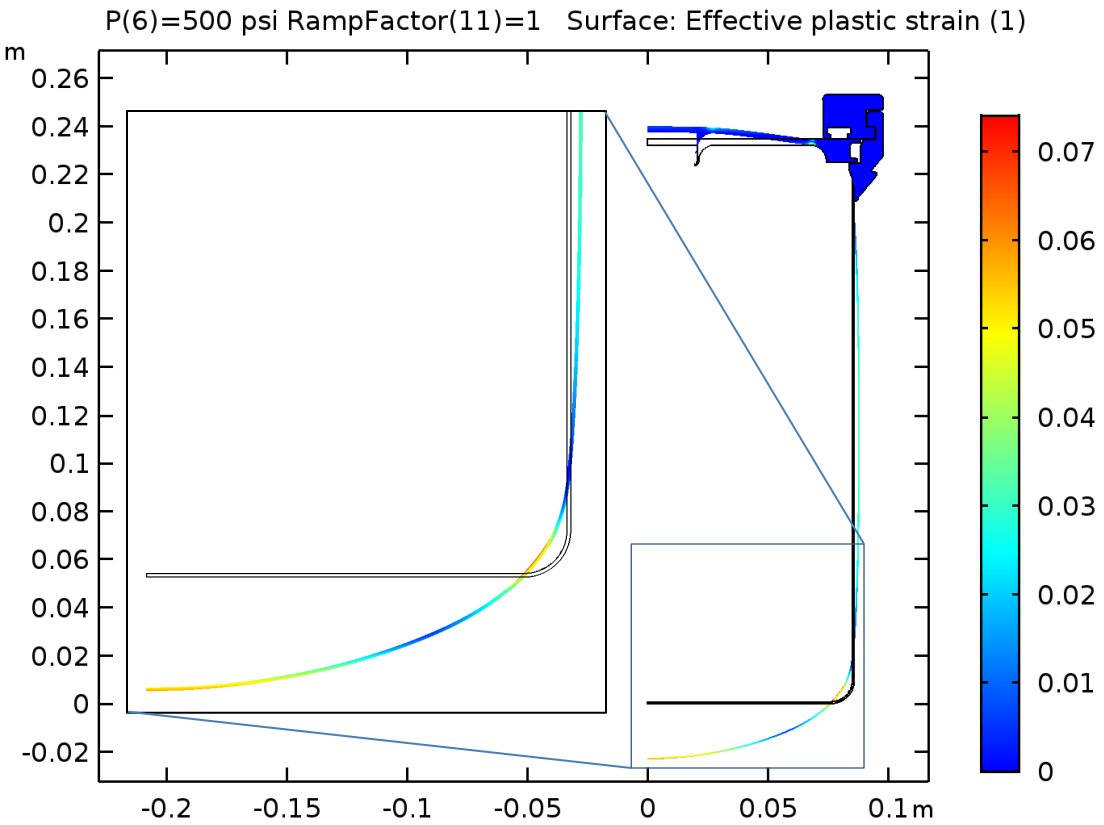
Results: 200 PSI

- Upper lid deflects ~2 mm
- Base deflection ~13 mm
- Base is also now in a “stretching” mode
- All plastic strains still less than 1%



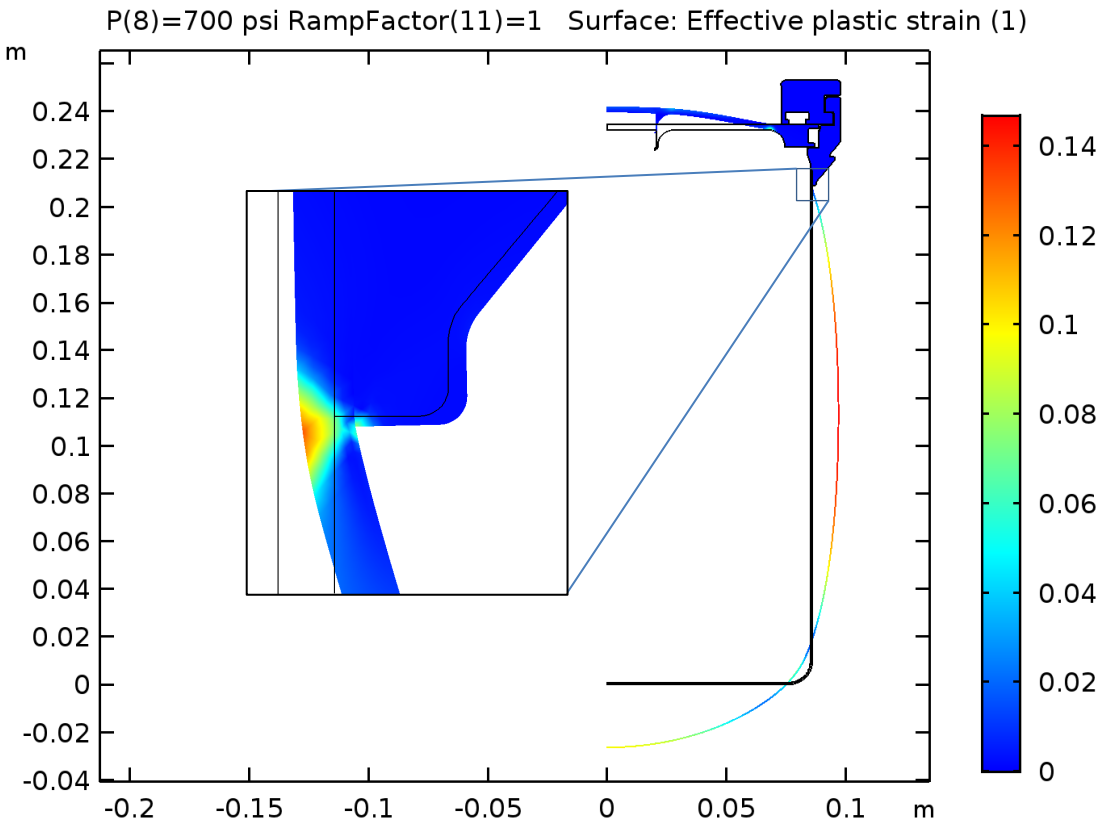
Results: 500 PSI

- Vertical walls of can are now deforming plastically
- Localized yield at root of lid
- Upper lid deflects ~6 mm
- Base deflection ~22 mm
- Wall deflection ~ 2-3 mm



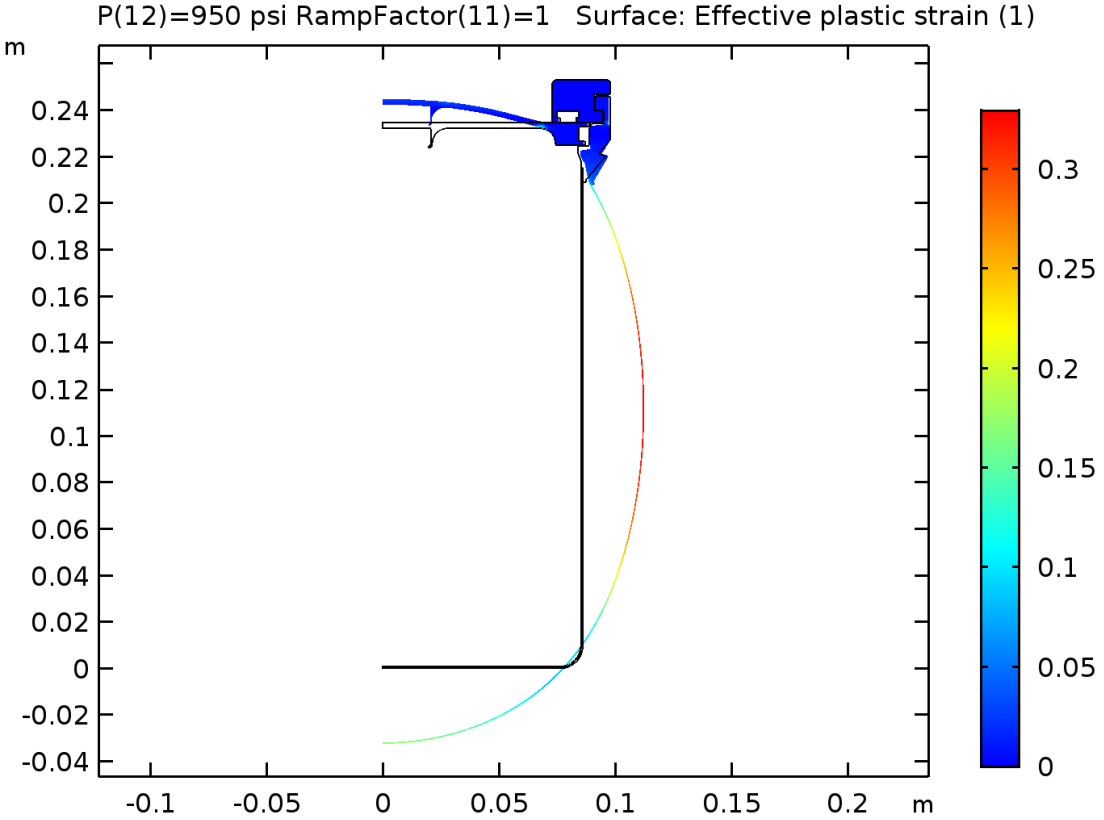
Results: 700 PSI

- Upper lid deflects ~8 mm
- Base deflection ~26 mm
- Wall deflection ~ 12 mm
- Wall is now expanding faster than lid or base
- Due to radial expansion and to plastic hinge at body/ring junction (inset)
- Max plastic strain is in middle of wall (~15%)

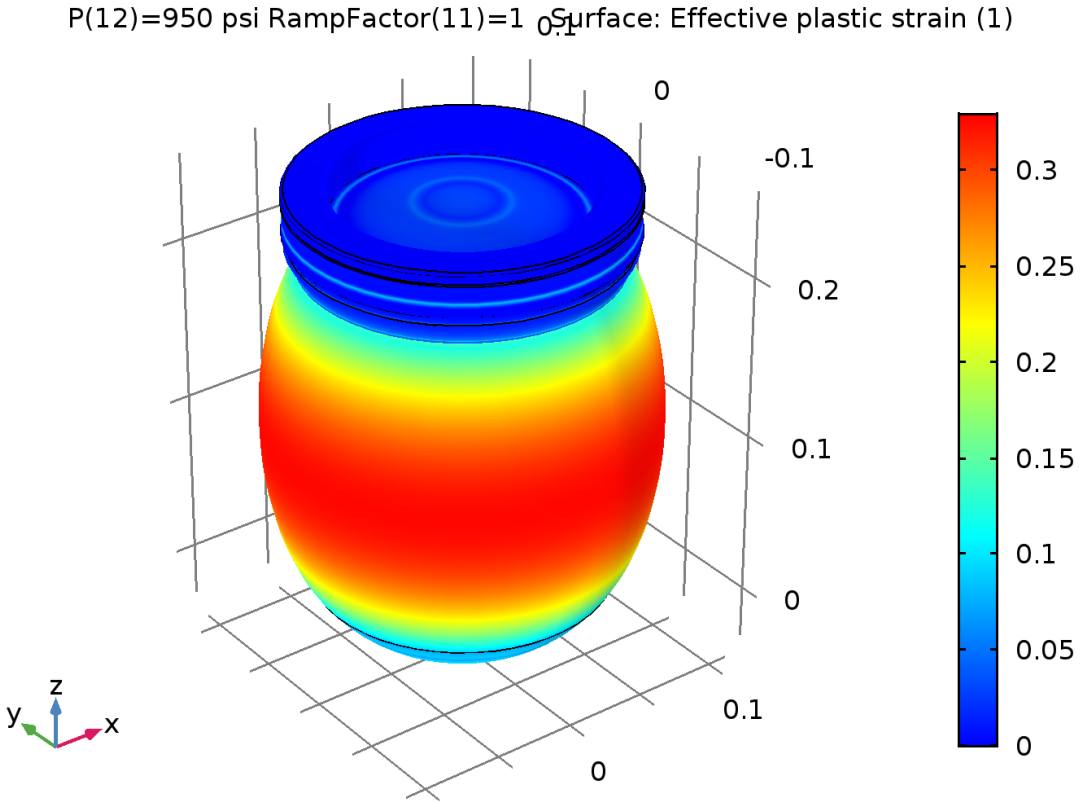


Results: 950 PSI

- Max plastic strain is in middle of wall (~33%)
- Wall is just below failure strain
- Upper lid deflects ~10 mm
- Base deflection ~31 mm
- Wall deflection ~ 27 mm



Results: 950 PSI



O-ring Failure

- High pressure can lead to extrusion of the o-ring into the gap near the gland
- Our initial gap is 0.5 mm which puts us close to the extrusion limit for the pressures we expect to cause mechanical failure
- Will update based on gap in deformed geometry
- Possible that extruded ring maintains seal
- Need durometer (Viton V-2 not specific)

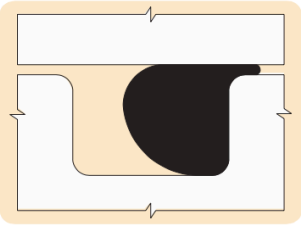


Figure 1-6: O-Ring Extruding

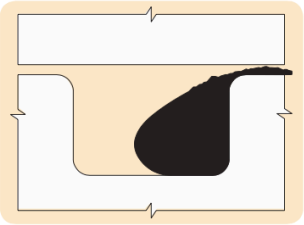
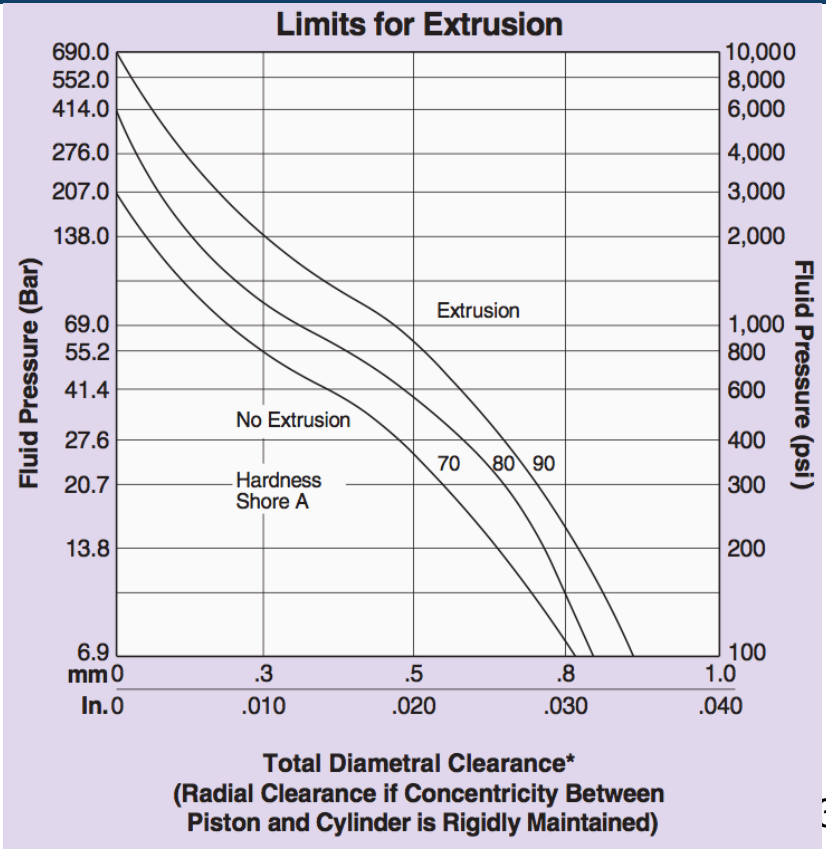


Figure 1-7: O-Ring Failure



O-ring gas permeation

- Earlier discussions did not lead to an agreed upon hydrogen generation rate
- Approach the problem based on the expected permeation rate as a function of pressure
- Need o-ring specs
  - Standard ”dash” number
  - O-ring lubrication?
  - Supplier if possible for details on specific chemistry

$$L = 0.7\, F\, D\, P\, Q\, (1-S)^2$$

where

L = Approximate leak rate of the seal, std. cc/sec.

F = Permeability rate of the gas through the elastomer at the anticipated operating temperature, std. cc cm/cm² sec bar (Many of these permeability rates are listed in Table 3-18, found at the end of this section)

D = Inside diameter of the O-ring, inches.

P = Pressure differential across the seal, lb/in²

Q = Factor depending on the percent squeeze and whether the O-ring is lubricated or dry (from Figure 3-11)

S = Percent squeeze on the O-ring cross section expressed as a decimal. (i.e., for a 20% squeeze, S = .20)

Replacement of PVC: Aging of Aromatic Polyurethanes Bags for Nuclear Storage

Joseph H. Dumont, Samantha J. Talley, Eamonn Murphy, Drew Geller, Zachary R. Brounstein, Kwan-Soo Lee, Andrea Labouriau

August 20th 2019

UNCLASSIFIED

Outline

- What happens to current sPVC bags in storage
- The long term aging of two alternatives: aromatic polyurethane bags (ester *vs*. ether)
- Summary

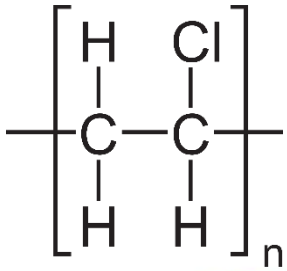
Acronyms

- sPVC: suspension polyvinyl chloride
- APU-ether: aromatic polyether urethane
- APU-ester: aromatic polyester urethane

What's the purpose of the bag? What is PVC?

- The role of the plastic bag is to protect workers who handle nuclear material from exposure due to loss of containment of stored materials

- Polyvinyl chloride (PVC) is a widely produced rigid plastic.
- PVC can be made flexible by incorporating plasticizers.
- Degradation of PVC occurs via dehydrochlorination (formation of HCl)



Polyvinylchloride (sPVC) bags and how they age in storage

<u>In storage, under temperature and/or radiation:</u>

- **Generation of HCl (dehydrochlorination)**
  - Corrosion of SAVY containers
- **Loss of plasticizer**
  - Change in mechanical, thermal, chemical properties

Aging of sPVC and Polyurethane (APU) bags

Bags for the storage and transport of nuclear material

- **Plasticized polyvinyl chloride (sPVC)**
- **Aromatic Polyether Urethane** APU (Ether)

→

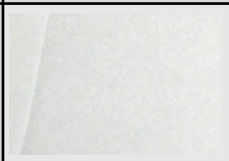
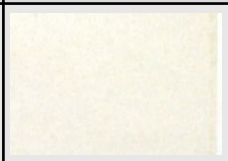
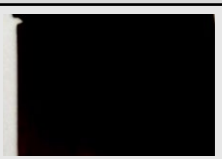
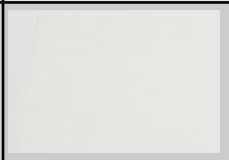
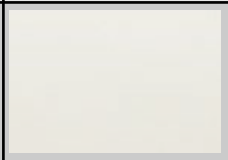
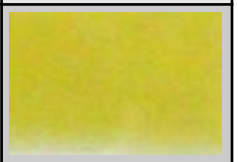
Aging study

- **Thermal aging -** *135°C , 10 days*
- **γ radiation –** *200 kGy*
- **α radiation –** *$5x10^{13}$ and $1x10^{14}$ cm^{-2}*

→

Characterization

Chemical	Thermal	Mechanical
FT-IR, NMR Contact angle Solvent swelling etc.	*TGA DSC*	*Tensile strength*

Samples	Pristine	Gamma irradiated	Thermally aged	Alpha irradiated	
Aging conditions	N/A	200 kGy	10 days at 135 °C	5.24 MeV, $5x10^{13}$ cm^{-2}	5.24 MeV, $1x10^{14}$ cm^{-2}
sPVC					
APU-ether					
316L coupon				No coupon placed in the alpha exposure	No coupon placed in the alpha exposure

Important changes are visible:

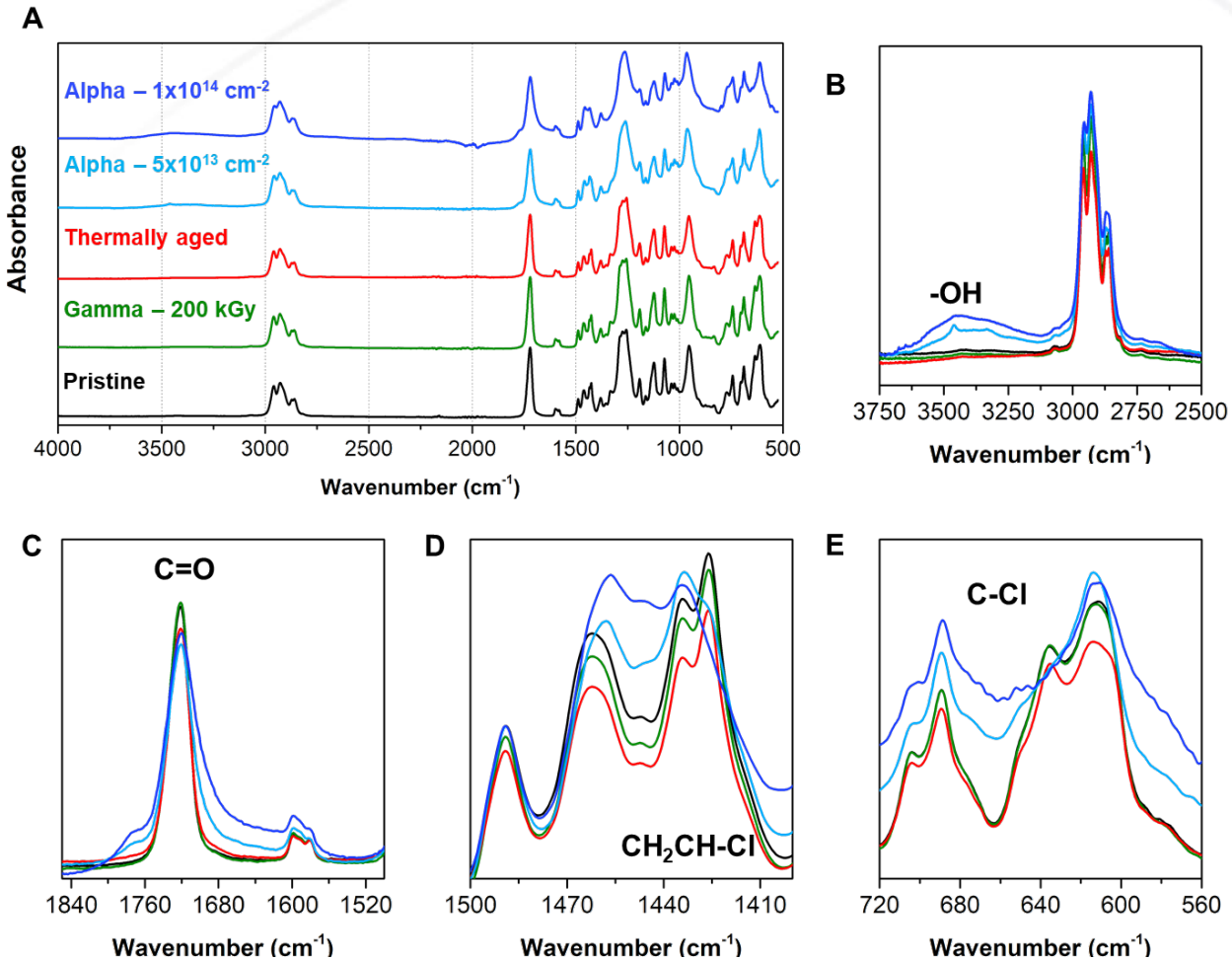
sPVC:

- darkening
- smell of chlorine
- plasticizer leaching out
- Corrosion of the SS coupon

APU:

- Yellowing of the material under alpha radiation.
- No corrosion, smell, plasticizer identifiable.

FT-IR spectra of pristine, thermally aged and irradiated sPVC



Important changes are visible:

- **3300 cm^{-1}:** ↑ OH intensity → degradation byproducts from the DOP
- **1720 cm^{-1} , 1780 cm^{-1} and 1640 cm^{-1}:** ↓ C=O intensity → loss of plasticizer. ↑ (C=O)-Cl and (C=O)-OH intensity → formation of chlorinated hydrocarbons
- **1460 - 1430 cm^{-1}:** changes in -CH_2CHCl- angular deformation,
- **720 - 580 cm^{-1}:** changes at the C–Cl stretching bands.

Summary: sPVC

- **Chemical properties:** sPVC → Strong evidence of degradation
- **Thermal properties:** sPVC degrades at 230°C
- **Mechanical properties:** sPVC → stiffer due to the loss of plasticizer
- **Other results:**

→ ^{1}H NMR analysis of the droplets confirmed the presence of diisodecyl phthalate (DOP).

→ sPVC contains up to 40 wt% of plasticizer.

→ Hydrophobicity of sPVC decreases when aged.

Dumont, Joseph H., Eamonn Murphy, Drew Geller, Kwan-Soo Lee, and Andrea Labouriau. "Effects of thermal aging and ionizing radiation on sPVC and aromatic polyether urethane used to store nuclear materials." *Polymer Testing* 78 (2019): 105960.

Aging study on polyurethane bags

Alternative bags for the storage and transport of nuclear material

- **Aromatic Polyether Urethane** APU (Ether)
- **Aromatic Polyester Urethane** APU (Ester)

→

Long term aging study

- **Thermal aging -** *85°C*
- **γ radiation -** *2 rad/h*
- **Thermal aging + γ radiation -** *85°C, 2 rad/h*

→

Time

- 3 months
- 6 months
- 12 months
- …

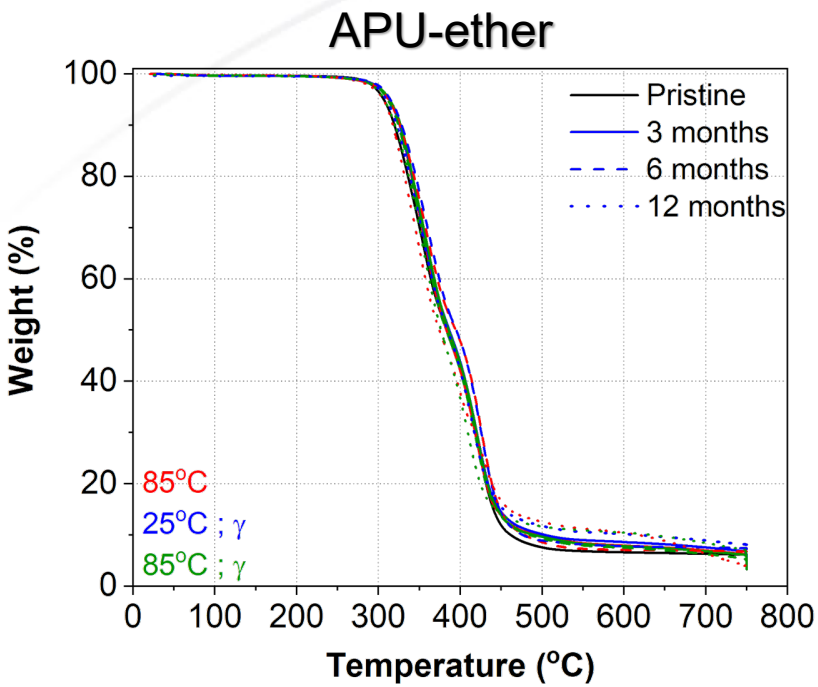
→

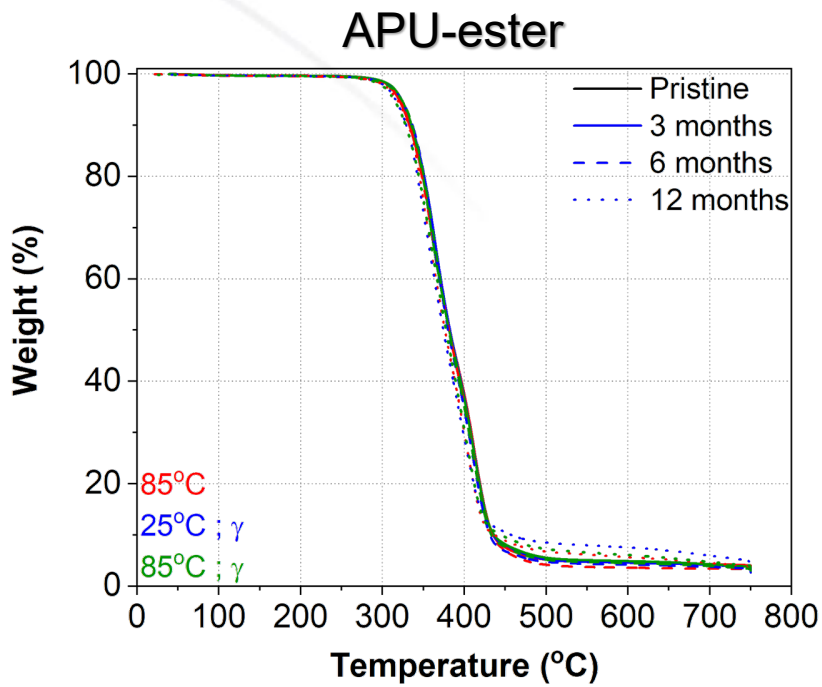
Characterization

Chemical	Thermal	Mechanical
FT-IR, NMR Contact angle Solvent swelling	*TGA DSC*	*Tensile strength Puncture test*

- Unique combination of aging conditions (low dose irradiation facility at Sandia Nat. Lab.)
- Understand the aging mechanism
- Ensure compatibility of the materials
- Predict the long term behavior under storage

TGA of aged bags

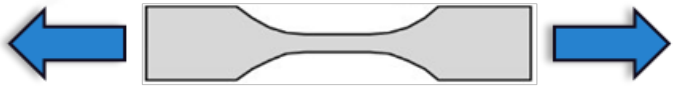




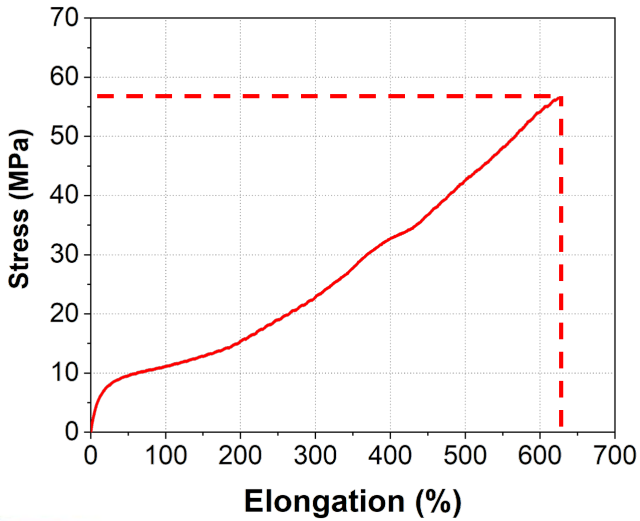
No significant changes in thermal properties

Mechanical testing – tension and puncture tests

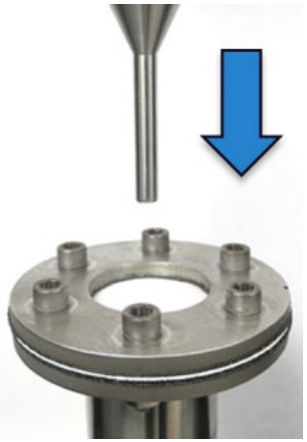
- Tension tests



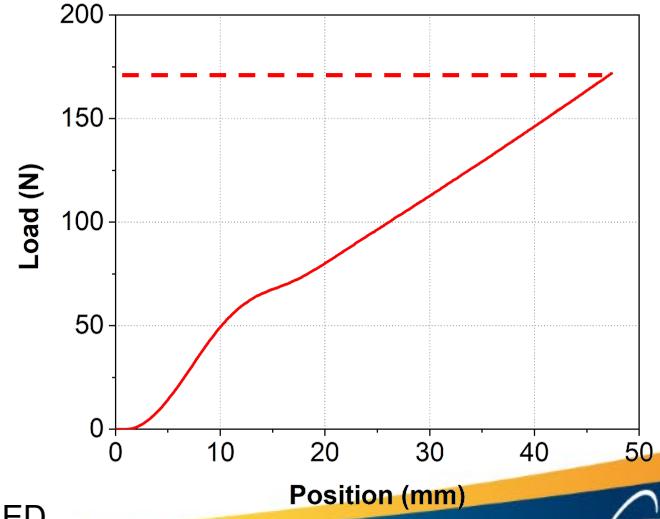
Conditions: **ASTM D412 (Die D)**
- Average of 3 Samples
- Sample Width: 3 mm
- Testing Temperature: 25 °C
- Testing Speed: 3.84 mm/s



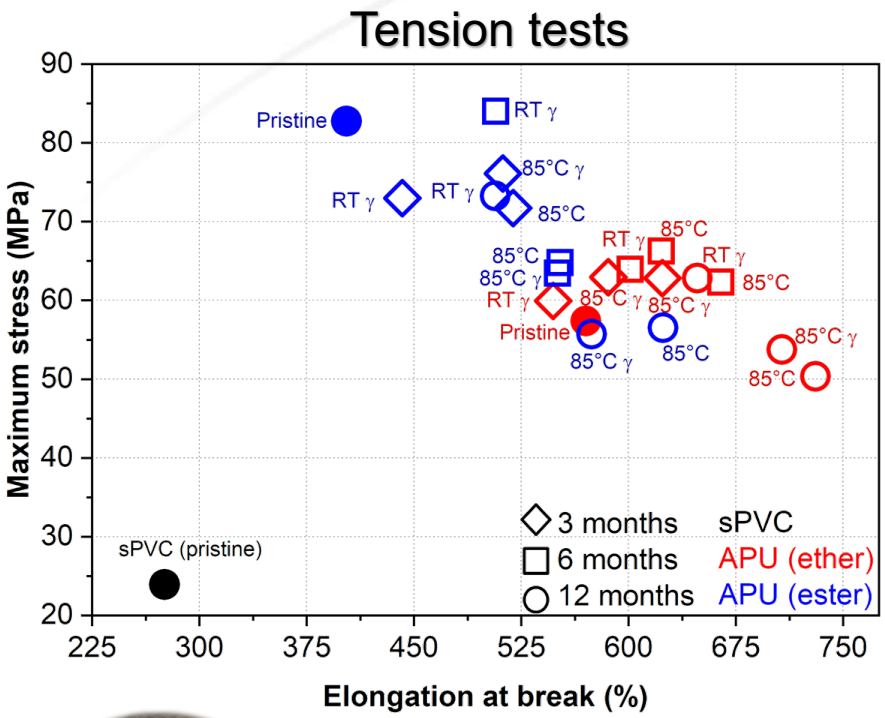
- Puncture tests

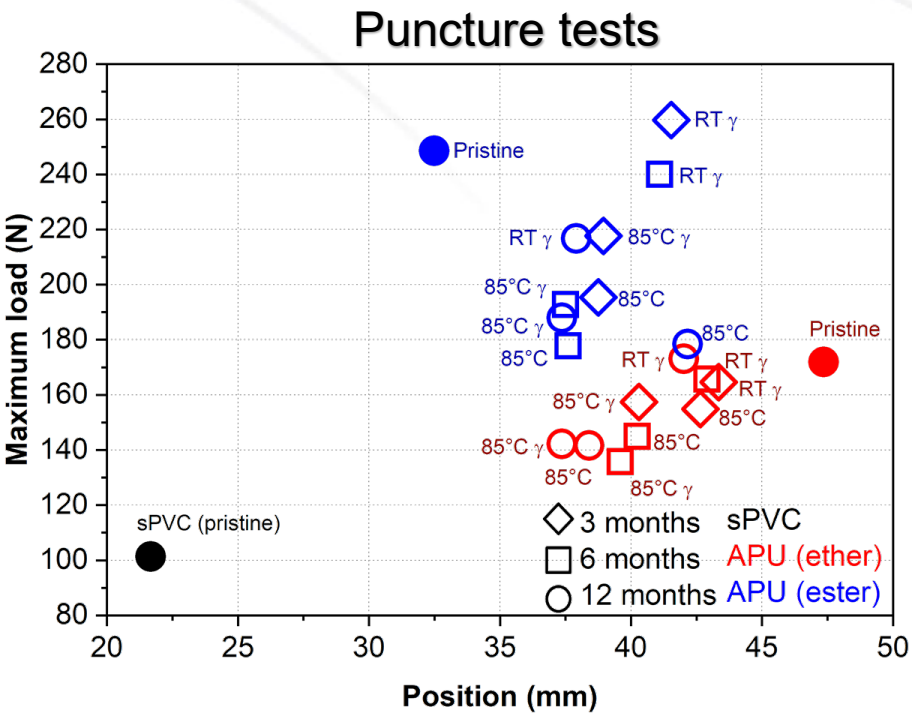


Conditions: **ASTM D4833**
- Average of 3 Samples
- Testing Temperature: 25 °C
- Testing Speed: 5 mm/s



How do these results compare to PVC?





The two polyurethane bags get **softer** when aged, unlike sPVC that gets **brittle**.

The mechanical properties outperform PVC bags in all areas

Summary: APU-ether *vs*. APU-ester

- **Chemical properties:** APU-ether and ester properties remain unchanged after 12 months.
- **Thermal properties:** properties remain unchanged. APU bags degrade at 320-330°C (*vs.* sPVC 230°C)
- **Mechanical properties:** Both materials get softer. Changes are more pronounced in APU-ester. Both materials outperform sPVC

Ongoing work will complement these results: head space analysis of the decomposition gases and NMR

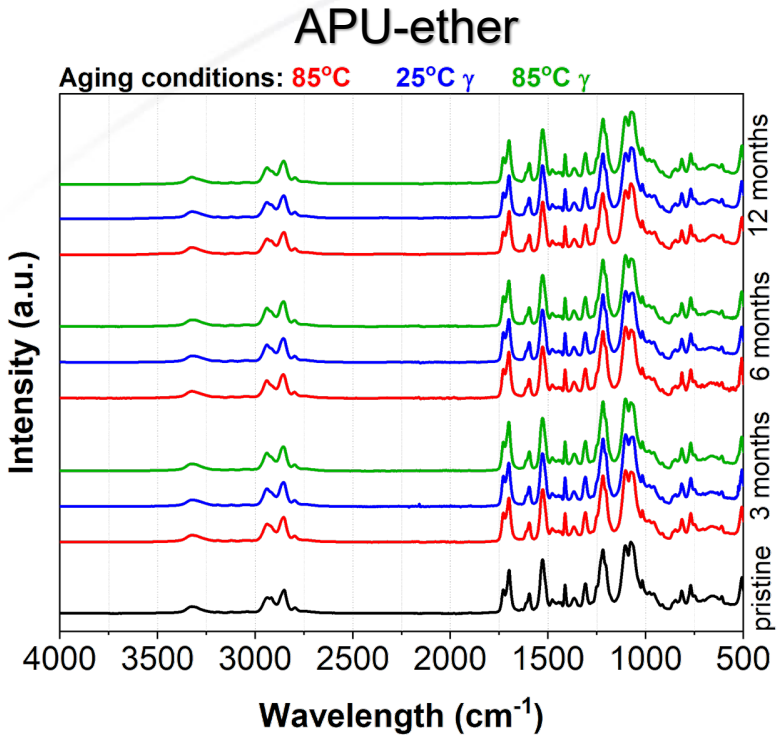
Acknowledgments

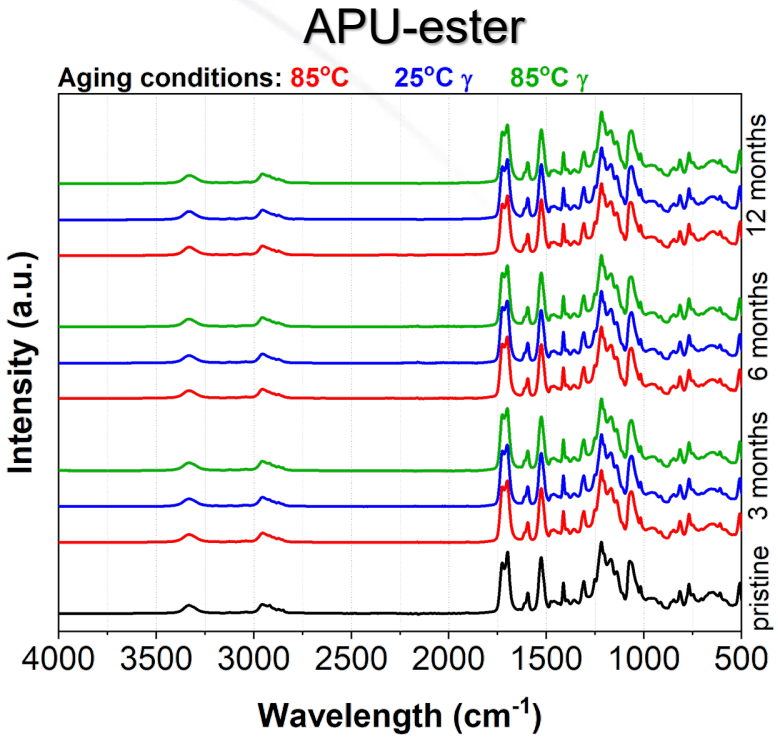
This work was performed under the US Department of Energy's National Nuclear Security Administration contract DE-AC52-06NA25396.

- Tristan M. Karns
- Timothy A. Stone
- Paul H. Smith
- Anna Carrol
- Jianchao Zhao
- Shaylynn Crum

Thank you

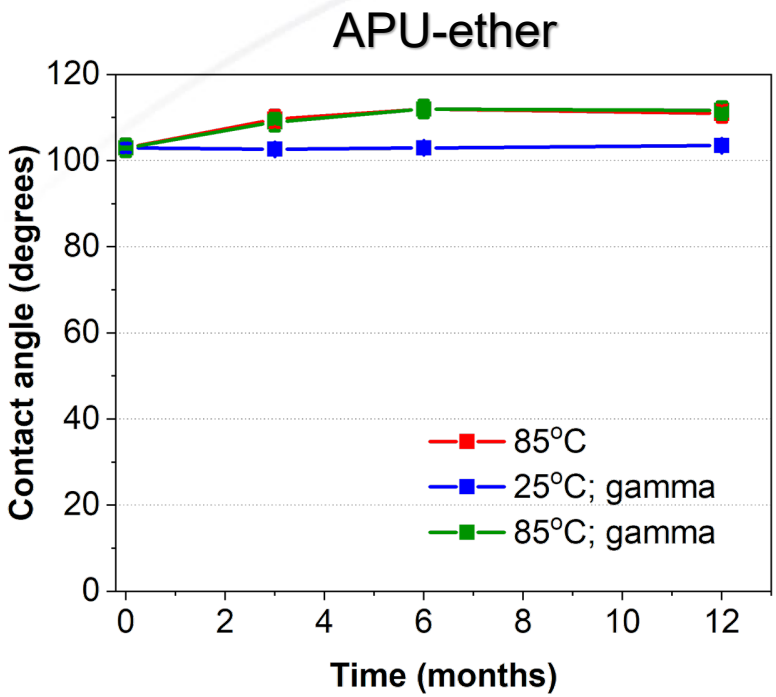
FT-IR spectra of aged bags

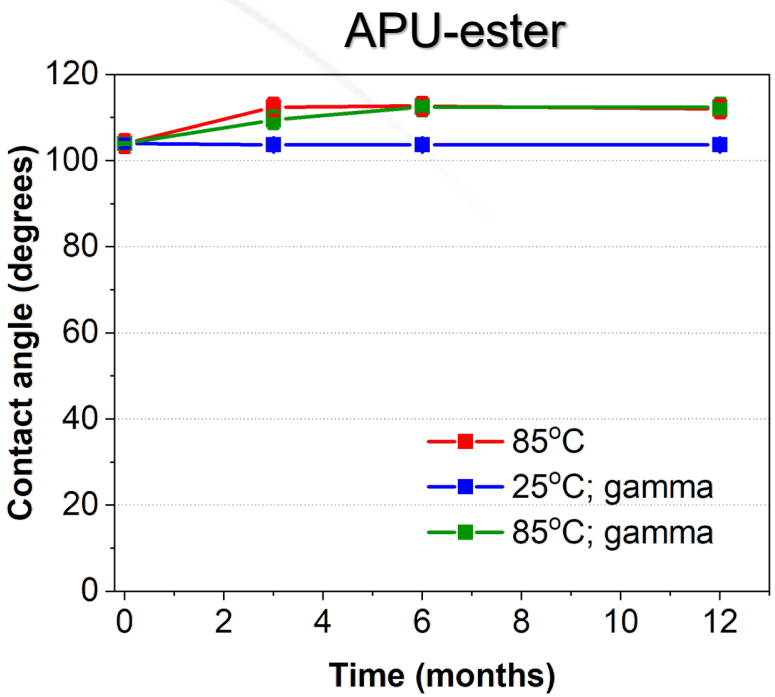




No significant changes in FT-IR spectra

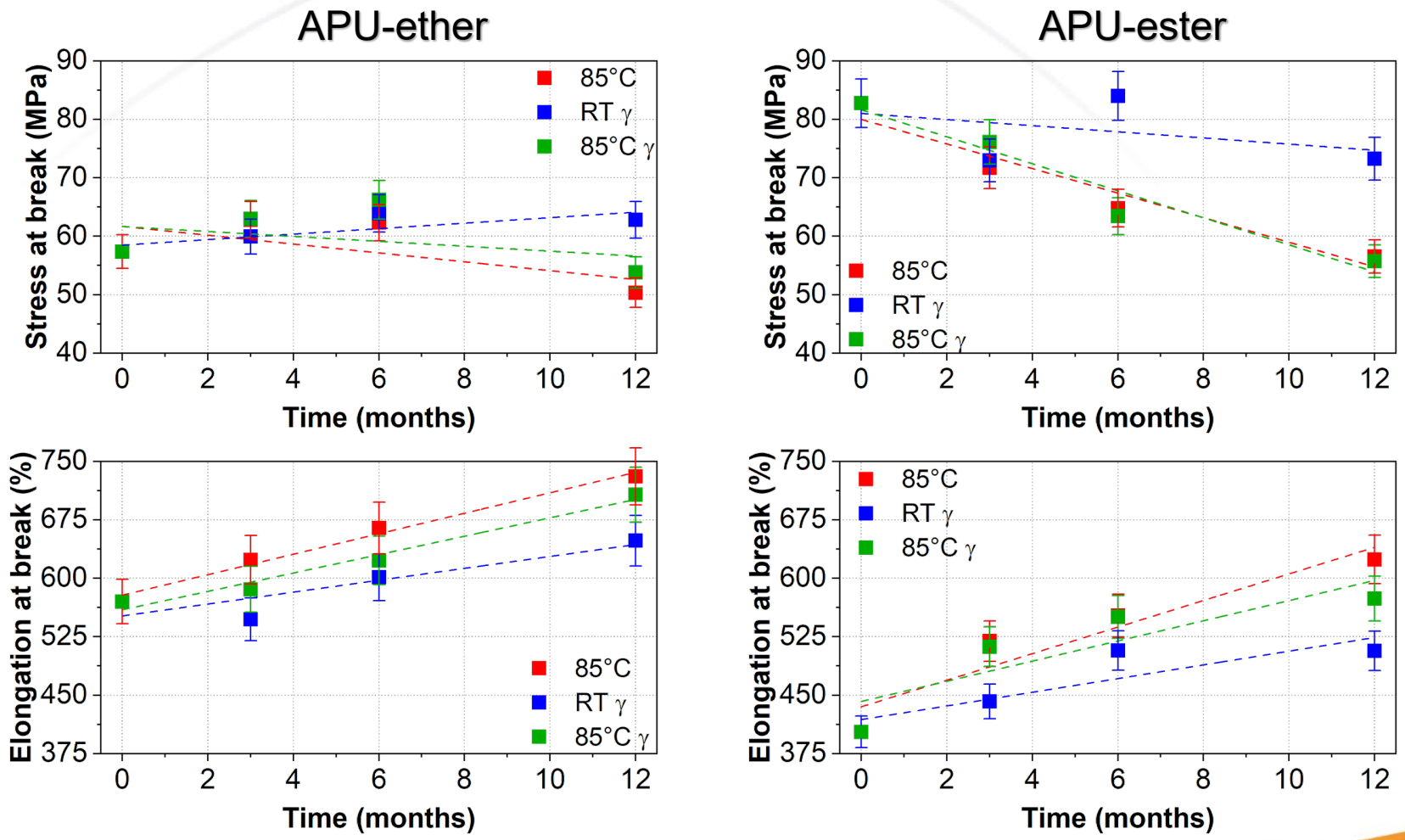
Contact angle of aged bags





Slight increase in contact angle after aging at high temperature → more hydrophobicity

Tension tests on aged bags

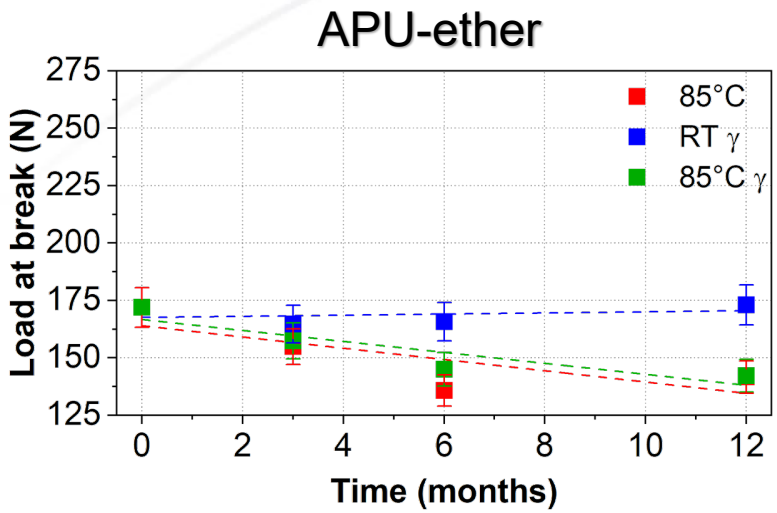


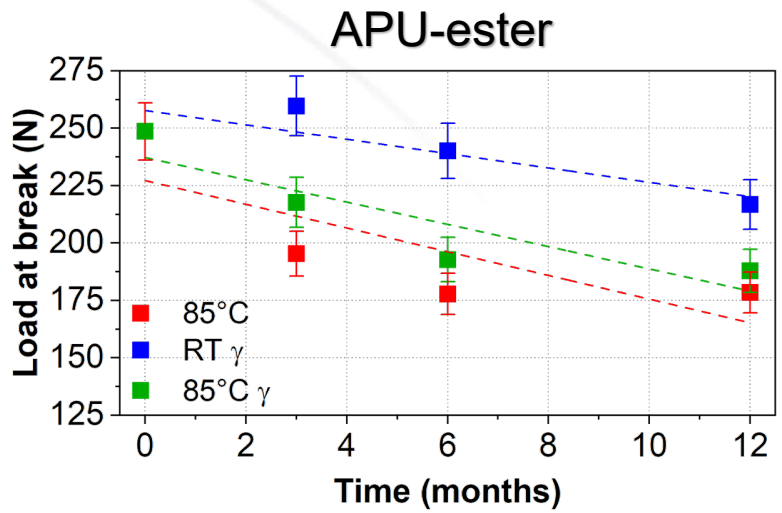
APU-ether: No changes in max stress, increase in max elongation (softening)

APU-ester: Slight decrease in max stress, increase in max elongation (softening)

Los Alamos NATIONAL LABORATORY EST. 1943

Puncture tests on aged bags





APU-ether: No changes in max load when aged at room temperature, slight decrease in max load when aged at 85°C

APU-ester: Slight decrease in max load when aged under all 3 conditions

Development and Application of the Modular Non-destructive Test System (MINTS) to Assess Corrosion Damage in Nuclear Material Storage Containers

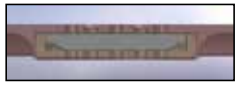
Rajendra Vaidya, Jonathan Gigax*, Adrian Abeyta, and Matthew Davenport
Strategic Programs and Engineering Directorate
*Materials Physics and Applications Division

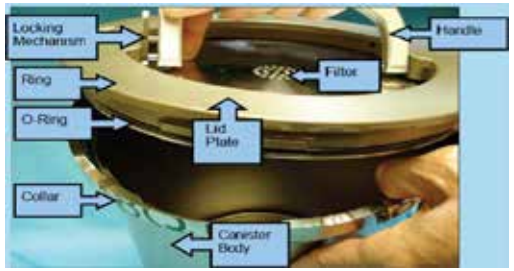
Nuclear Materials Packaging Program Review,
Los Alamos National Laboratory
August 20-21, 2019

Operated by TRIAD National Security for the U.S. Department of Energy's NNSA

Program Needs

LANL is the Design Authority on the SAVY Container for the NNSA





Program Needs and Current Issues

Goal: Continuous Validation of Design Life

Recent Issues: Corrosion Observations

Hagan Container

SAVY Container

Examining severity and mechanisms: surface erosion, pitting, stress corrosion cracking

Current Issues

Understand the extent and role of corrosion in a multivariate environment and its effects on mechanical properties and life of the SAVY containers

Research Summary

- Trade Study to Evaluate and Competing Non-Destructive technologies

Four different Techniques (Eddy Current testing (ECA), Ultrasonic testing (UT), Laser Shearography, and Fiber-optical technology) were evaluated as part of a detailed trade study. Various Technical and Operational factors were considered. *ECA and UT were the down-selected techniques.*

- Baseline thickness measurements on flat samples and pristine (new) containers and pit detection

Wall thickness measurements were completed using UT and ECA. Results were compared to those from a Micrometer and Contour Measurement Machine (CMM). *UT results were within 2.5% of the micrometer results and 5.5% of the CMM measurements.* The results are repeatable. A one tailed z-test calculated the mean difference between the values to be less than 0.0175 mm at a confidence interval of 99%. Ultrasonic testing gives you the ability to apply a go/no-go criteria to the inspection process. The minimum accurately measurable wall thickness is 0.25 mm. Below this value, the initial pulse was too close to the back-wall echo and the waveform changed dramatically rendering the measurements inaccurate. *ECA was not accurate for thickness measurements but can detect pits, cracking, and other flaws (down to 0.0762 mm).*

- Laboratory Corrosion Experiments

Container material Samples were subjected to various molarities of HCl (8, 10, 12M) and $FeCl_3$ (1.5 and 3M) to mimic extreme corrosion conditions and determine the feasibility of using the NDE techniques to detect wall thickness loss and pitting.

- Validation of Current SAVY containers

Containers currently in use were tested using the NDE tools. Results were compared to those obtained from other conventional measurement techniques such as a CMM. *UT can be used in lieu of CMM measurements.*

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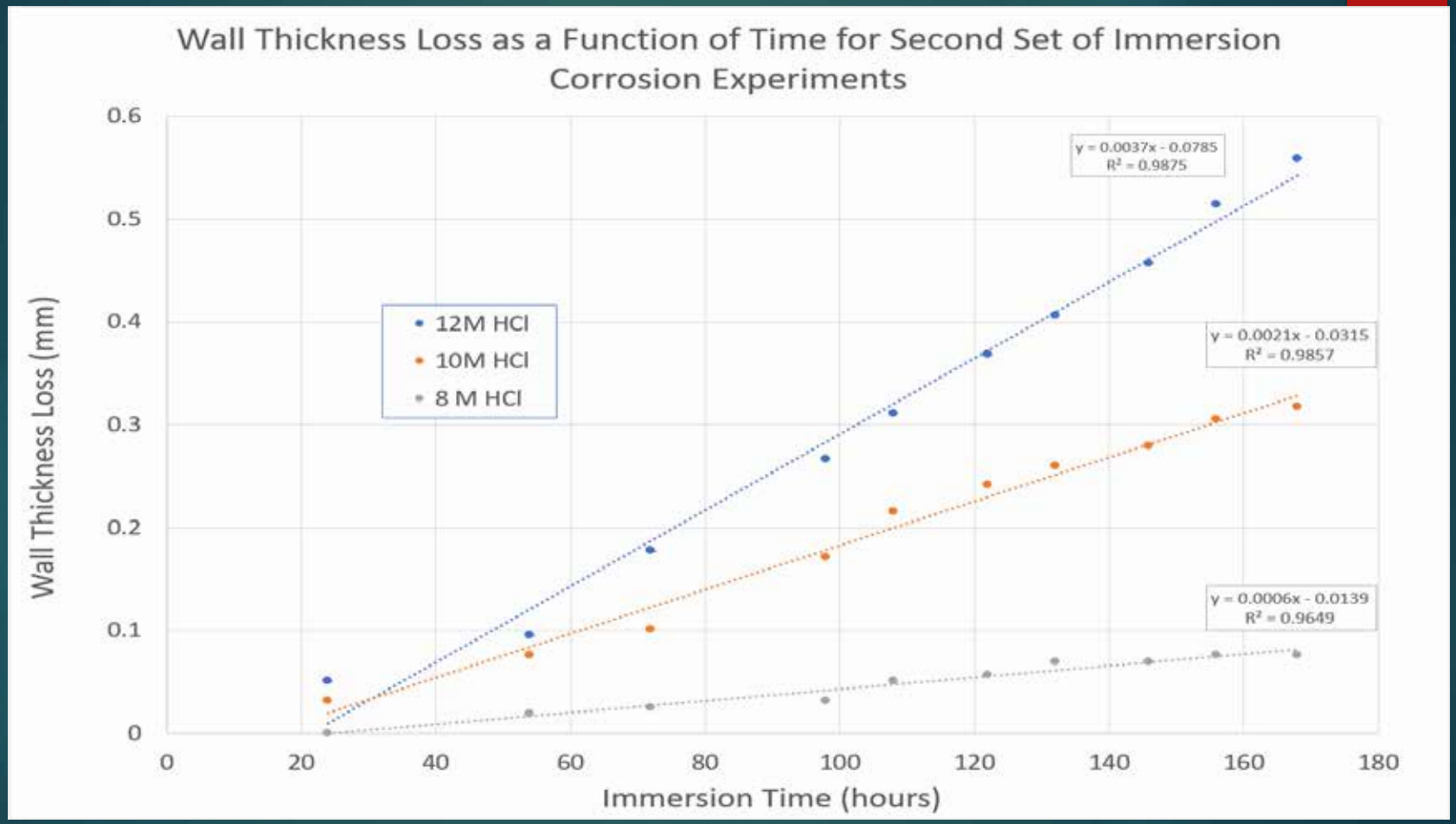
Equipment

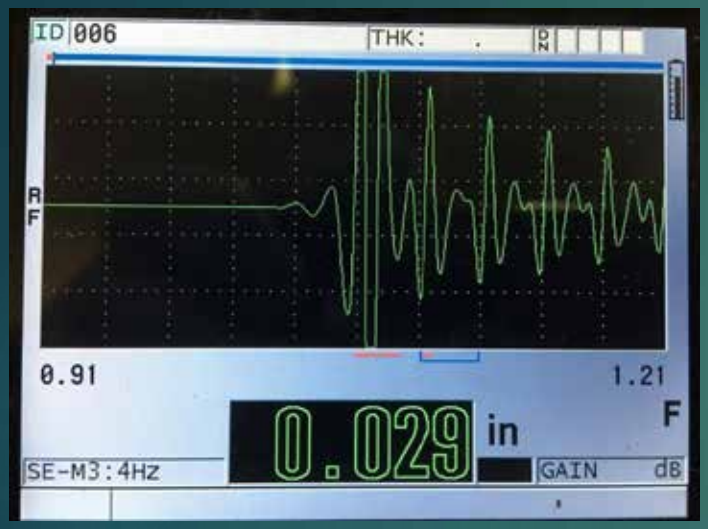
- Ultrasonic Technology (Olympus)
- 16 channel Focus PX
- 38DL-Plus Handheld Instrument
- 15 MHz Delay Line Sonopen Transducers

- Eddy Current Technology (Olympus and Zetec)
- OmniScan MX and MIZ-200-1
- 32 channel ECA probe
- ECA Semi-Rigid probe
- ECA Flexible probe

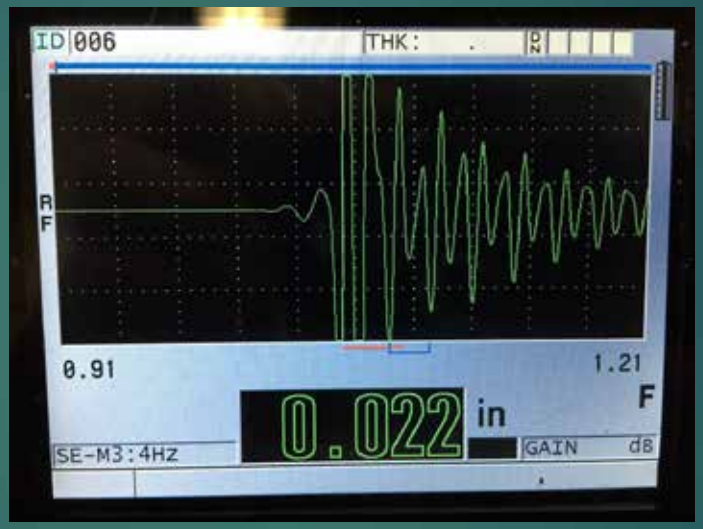
- Mechanical System
- Gantry system and Stages
- Ball bearings
- Electric motors
- Custom analysis software (LabView)


Wall Thickness Loss as a Function of Time for Second Set of Immersion Corrosion Experiments
Wall Thickness Loss (mm)
Immersion Time (hours)
0
0.1
0.2
0.3
0.4
0.5
0.6
0
20
40
60
80
100
120
140
160
180
12M HCl
10M HCl
8 M HCl
y = 0.0037x - 0.0785
R² = 0.9875
y = 0.0021x - 0.0315
R² = 0.9857
y = 0.0006x - 0.0139
R² = 0.9649

Waveform Data for sample exposed to 10M HCl



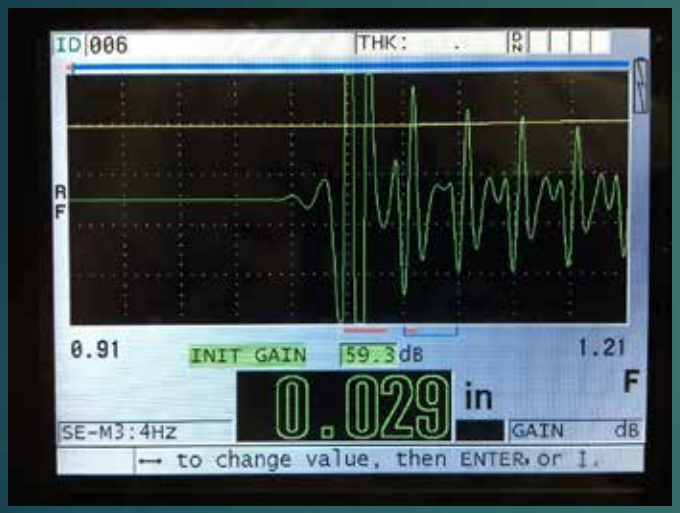
316L Control



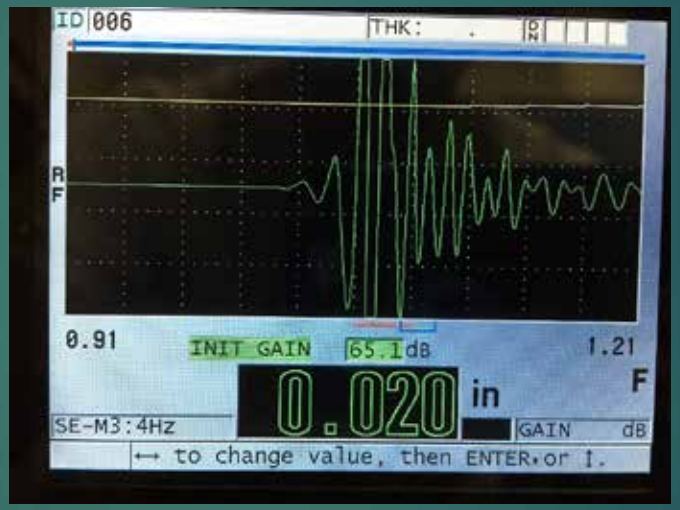
10M HCl for 7 days

Uniform Corrosion

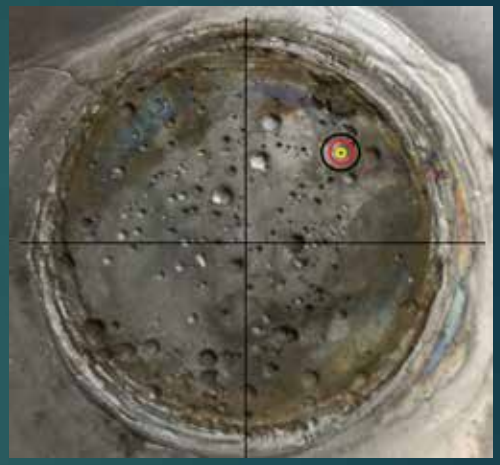
Waveforms for sample exposed to 1.5M $FeCl_3$



316L Control



1.5 M $FeCl_3$ for 48 hours



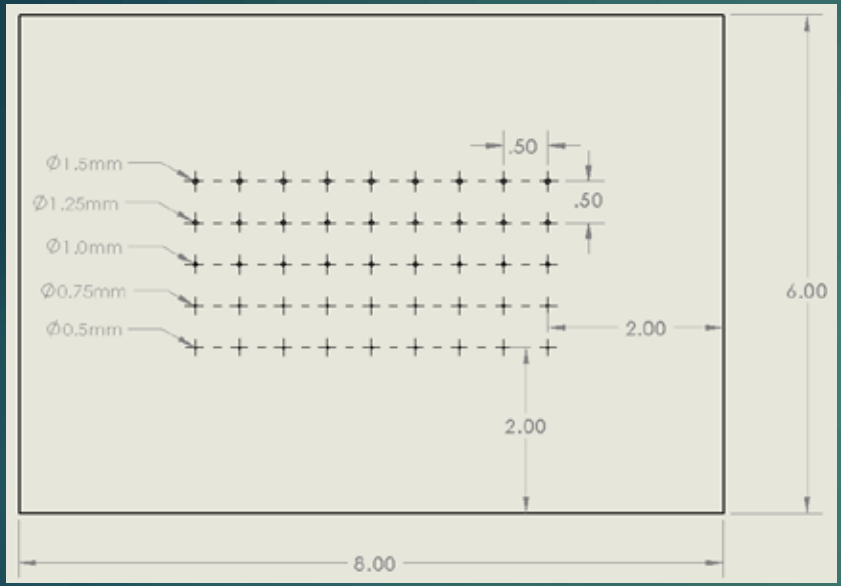
Pitting

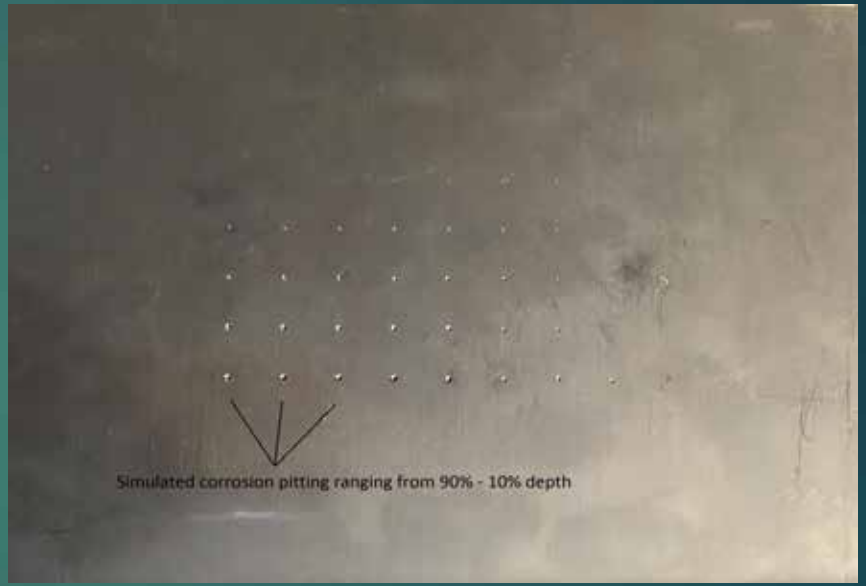
UT vs. CMM Data on containers extracted from the Surveillance Program

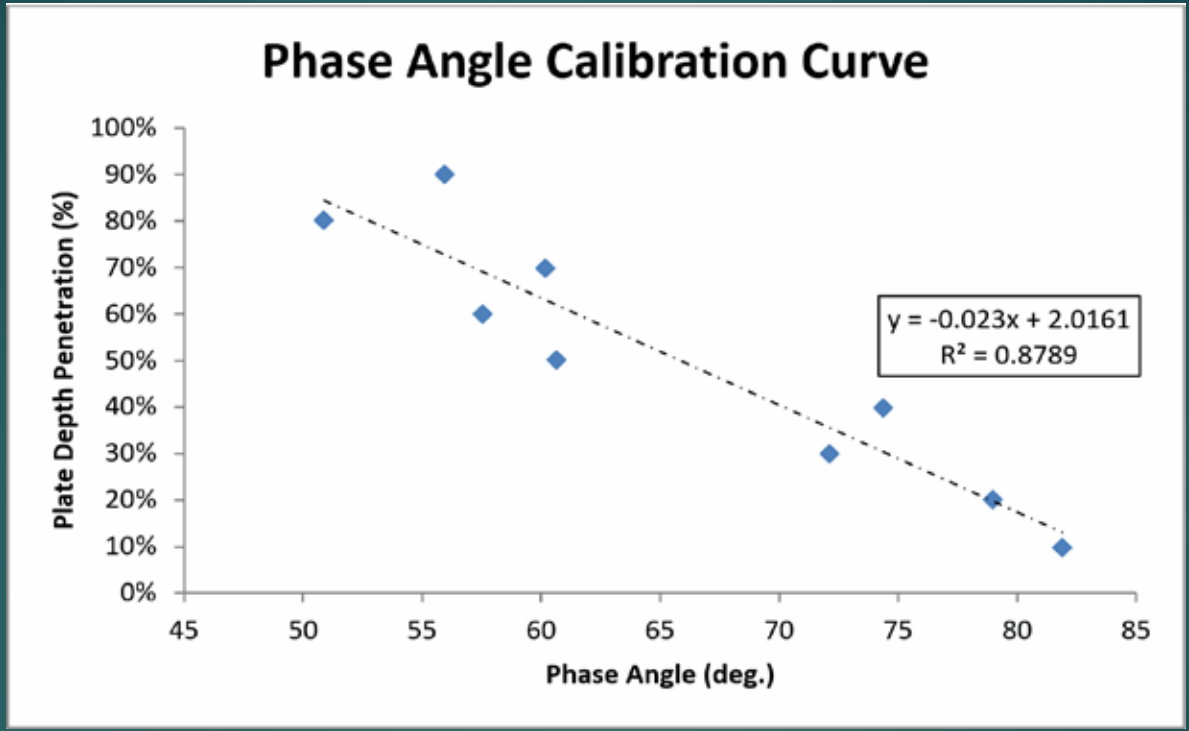
	Measurement on Height				Measurement on Base			
Serial #	UT Meas. (mm)		CMM Meas. (mm)		UT meas. (mm)		CMM Meas. (mm)	
	max.	min.	max.	min.	max.	min.	max.	min.
011305020B	0.762	0.56	0.813	0.67	0.61	0.56	0.602	0.592
081305070B	0.787	0.53	0.828	0.68	0.58	0.53	0.594	0.554
091205141B	0.787	0.56	0.803	0.68	0.58	0.53	0.607	0.533

Measurement	Greatest Relative Error (%)
Long. max.	6.3
Bottom max.	3.7
Bottom min.	5.6

Standard Machined Test Plate





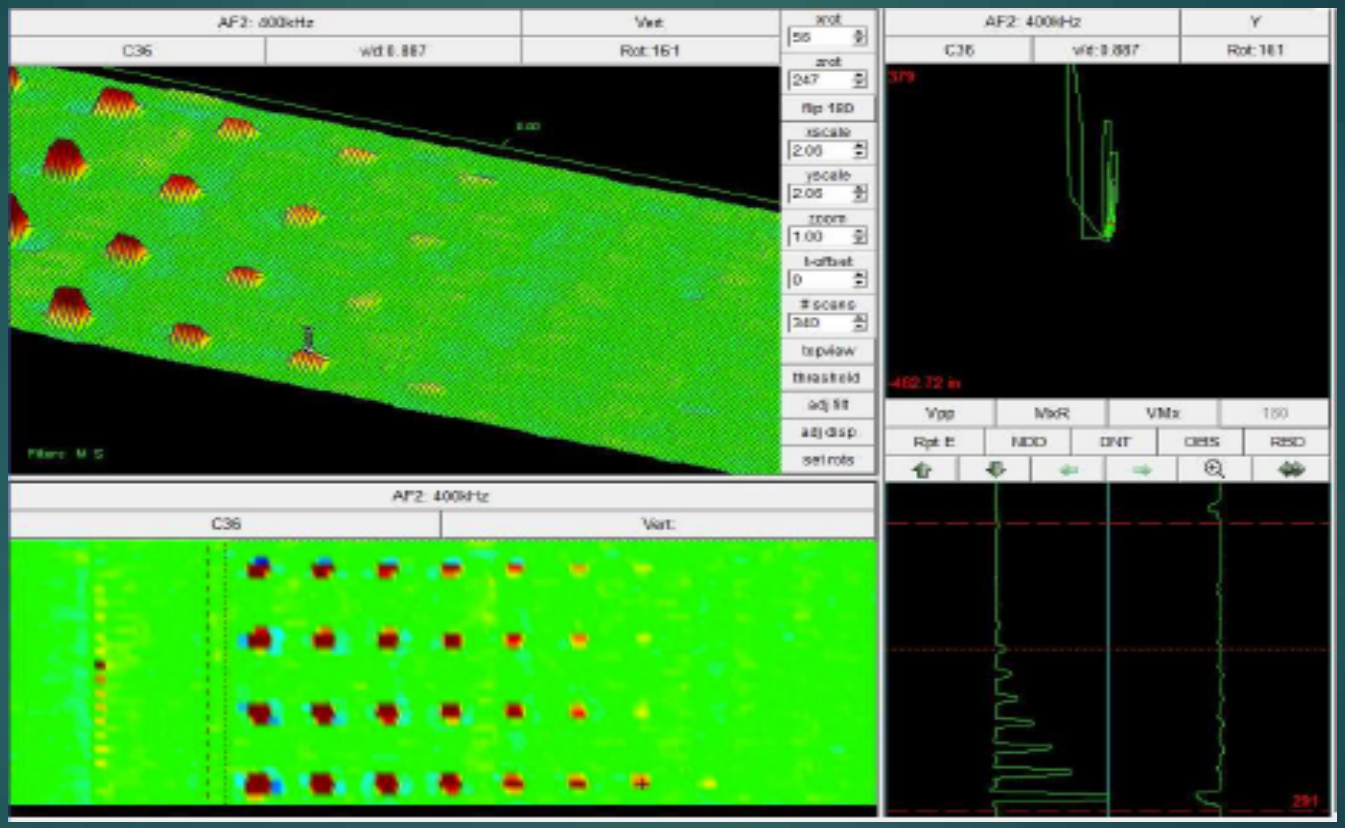


The measured phase angle plotted for each pit depth (10-90%) above. The phase angle is translated into pit depth below. The actual pit depths are from the machined test plate.

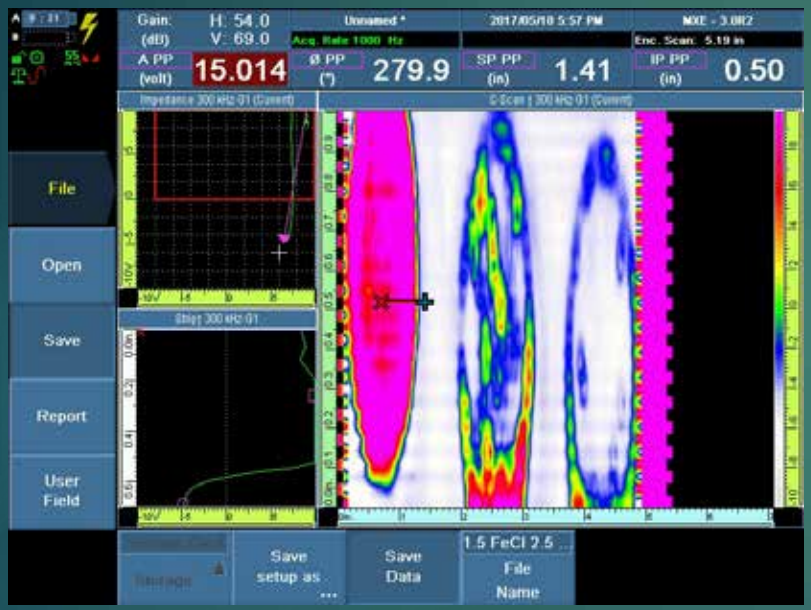
Actual versus Measured Depth on the Standard Machined Plate

Derogation Depth (%)	Test 1 (mm)	Test 2 (mm)	Test 3 (mm)	Actual Depth (mm)
90%	0.5565	0.5689	0.6001	0.6858
80%	0.644	0.6319	0.6290	0.6096
70%	0.4813	0.4567	0.4795	0.5334
60%	0.5285	0.5407	0.5161	0.4572
50%	0.4742	0.4881	0.4637	0.381
40%	0.2324	0.2042	0.2319	0.3048
30%	0.2725	0.2657	0.1920	0.2286
20%	0.1516	0.1499	0.1517	0.1524
10%	0.1008	0.0815	0.1359	0.0762

Mapping of standard test plate



Eddy current array (ECA)

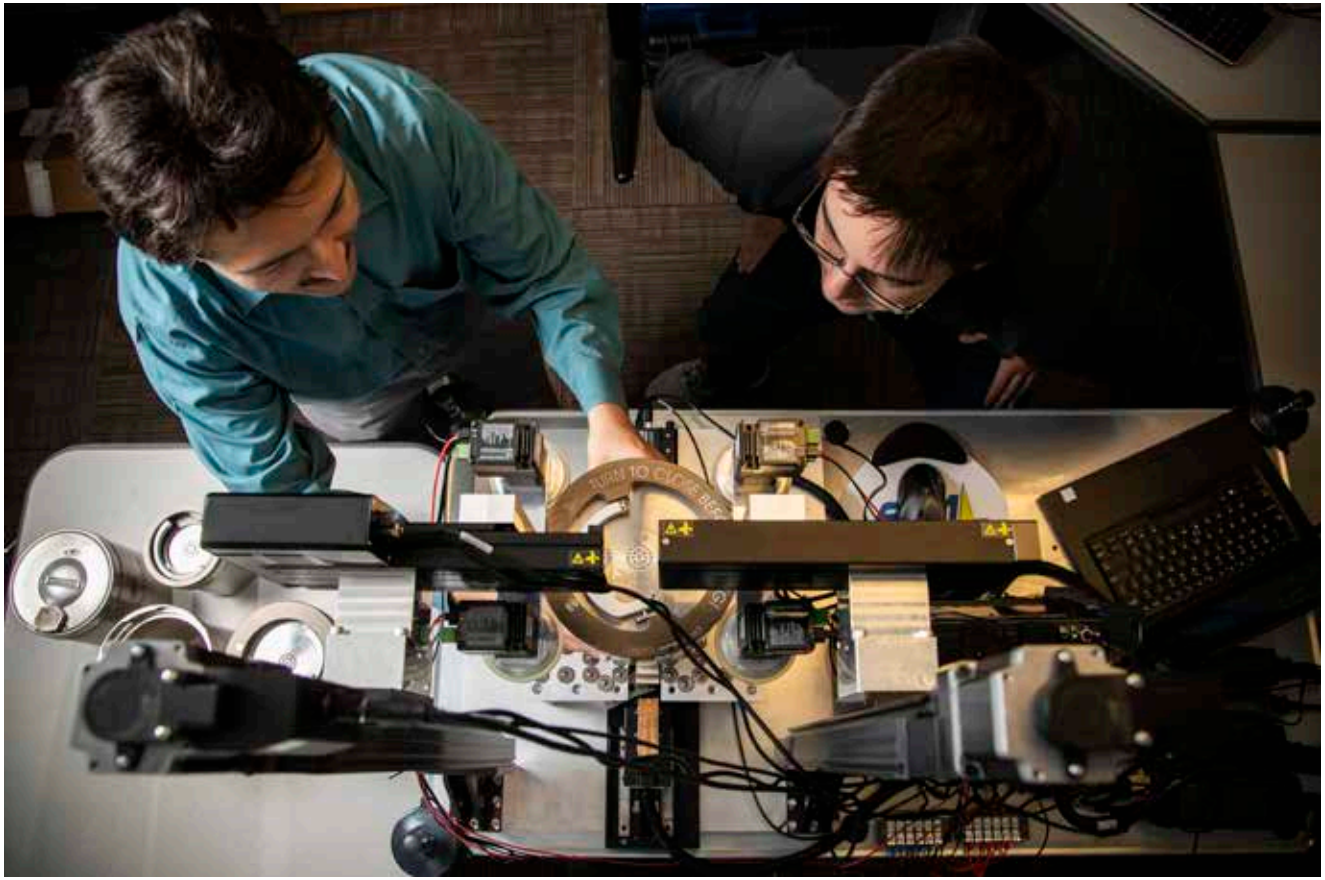


Omni Scan Mx C-Scan analysis of 316L exposed to 3M $FeCl_3$ at 7 days (Left) and 1.5M $FeCl_3$ at 7 days (Right)

Surface of 316L samples exposed to 3M $FeCl_3$ at 7days (Left) and 1.5 M $FeCl_3$ at 7 days (Right)

The MINTS (Modular Integrated Non-destructive Test Setup)

The MINTS Table



Current Status

Development of MINTS (Modular Non-destructive Test Setup)

New:

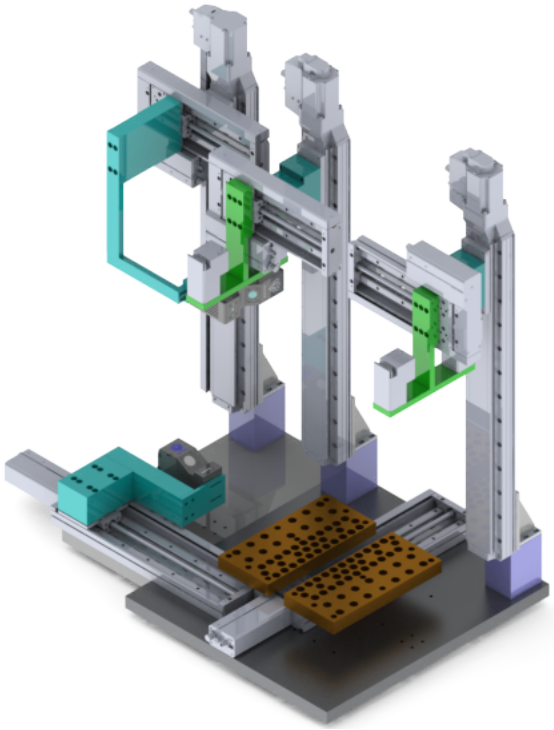
- First application of Non-destructive testing to assess the structural health of nuclear material storage containers *in-situ*.
- Combination of two complimentary test techniques onto a single integrated platform allows for a broader interrogation of corrosion processes.
- Ability to interrogate varying container sizes.
- Short test time durations (~10 mins.), Go/No-go decision making capabilities.
- Modular nature of the test setup design allows for the techniques to be applied in a variety of settings and the addition of other tools (laser interferometry).

Key Questions and Improvements for next phase

- Existing (COTS) eddy current system limited to a quick visual inspection routine.
  - Extracting and correlating data to container positions cumbersome and unreliable.
- Identify equipment necessary to drive coils at desired excitation frequency with a capability to be integrated with existing LabView software.
- Complete a detailed eddy current signal analysis and document in a signal library for future use.

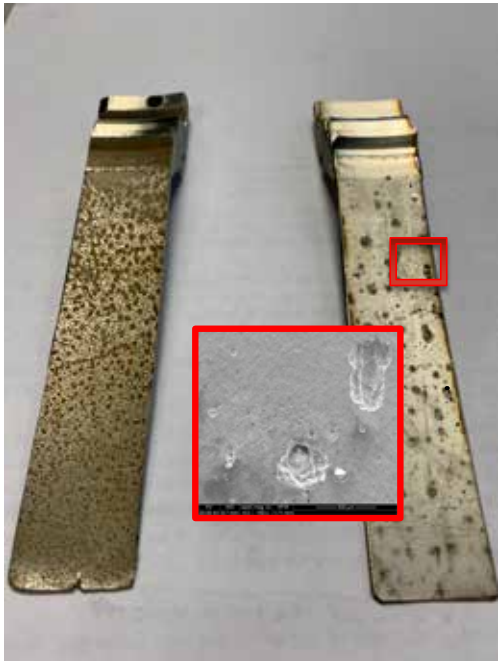
Key Questions and Improvements for the next phase

- Are external stresses an important factor to consider?
  - For existing corrosion, impacts may cause failure of wall material.
  - Dents on outer container wall translate to tensile stresses on inner wall. These enhance corrosion pitting/cracking!
- External surface profiling coupled with finite element modelling enables determination of stress without destructive interrogation.
- Meso/microscale mechanical testing will also be used to determine upper limit of external defect sizes (i.e. dents) that may lead to failure.

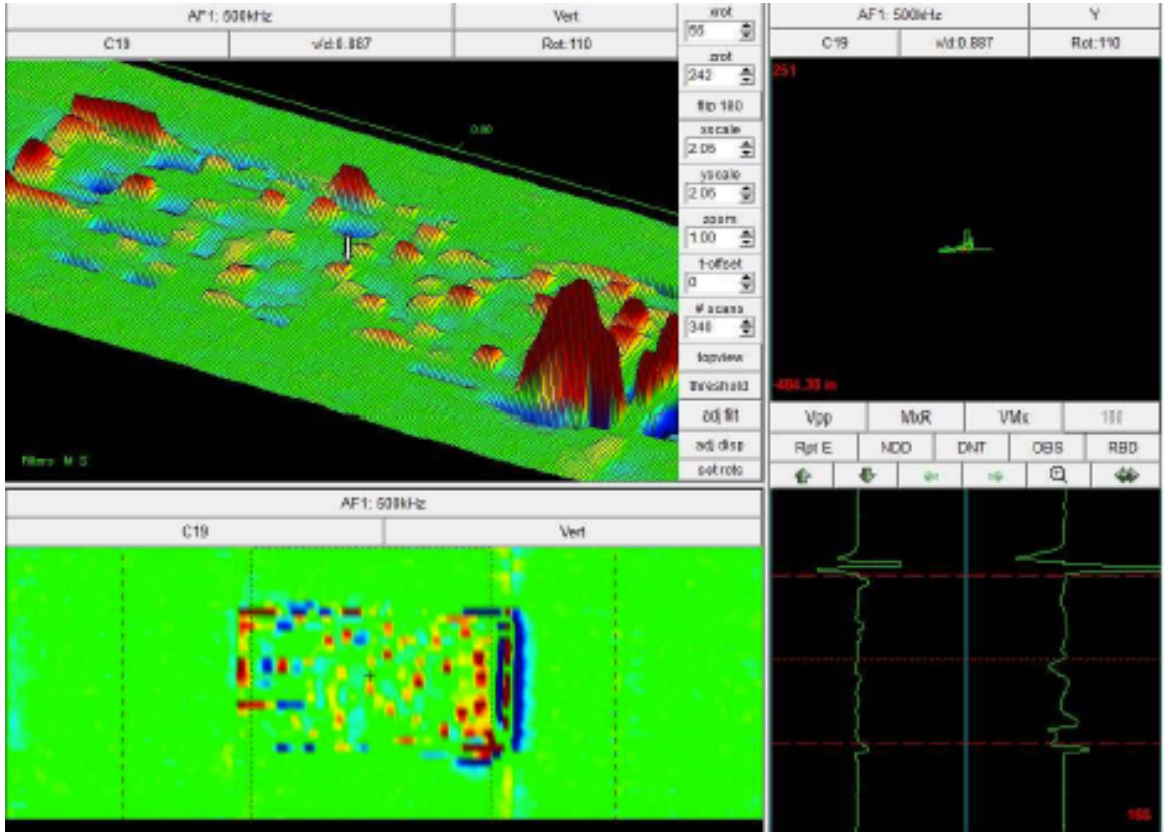


Motivation

- Why are these items of importance?
  - Developing predictive models for container service lifetime requires understanding key corrosion phenomena that contribute to container degradation.
  - Tracking pitting evolution in a single container reliably depends on a system that can accurately measure wall thickness losses and damage externally.
  - End goal is to be able to say with certainty when a container needs to be removed from service.



3-D Mapping of Corrosion Pits on the Inner surface



Roadmap for the next Phase

Develop improved eddy current detection techniques

- Integrate new equipment that features programmability for automated analysis.
- Design or modify existing detectors to follow contours and defects in container wall for more accurate assessments of pitting.
- Detailed eddy current signal cataloging.

Develop a high strain rate mesoscale mechanical testing system

- Design and assemble a mechanical tester capable of performing compression/tension loading at impact strain rates (i.e. $10^3\ s^{-1}$) for quantitative cataloging.

Develop stress analysis techniques

- Integrate surface profiling into system and develop software to construct 3D model of scan as an initial condition for finite element analysis

Surveillance of SNM Containers Using Mobile Robotics

LA-UR-19-25142

Cheryl Brabec, Cristy Abeyta,
Alexander Feldman, David Grow

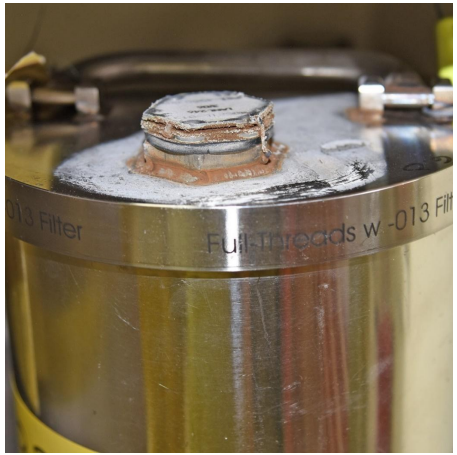
[Paper 330]

UNCLASSIFIED

Motivation

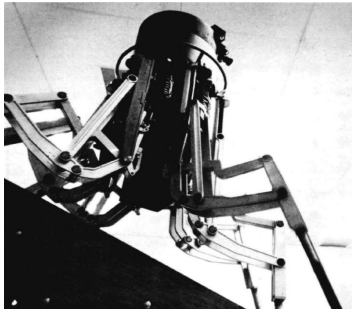
- Containers have to be robust and long-lasting
- Containers must nonetheless be monitored
- Manual inspection may entail significant dose
- Issues may only be salient only when once exposure risk reaches a significant level
- Robotics may:
  - Supplement current surveillance methods
  - Improve data collection
  - Reduce worker dose

Types of Container Issues



UNCLASSIFIED

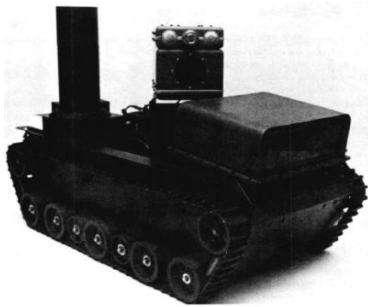
Rosa - core defueling



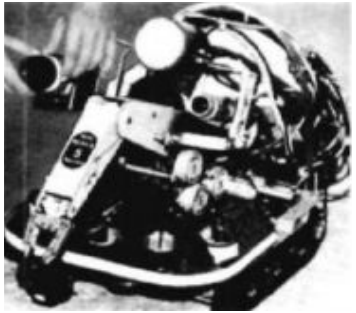
Odex - general walking robot

Fred - decontamination

Remote scabbler (decontamination)

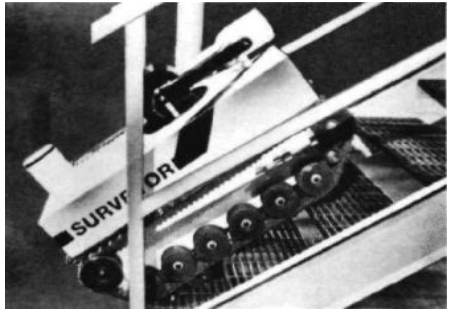


IRIS - general purpose robot for hazardous environs.



SISI - remote surveillance

Kluge - surveillance & equipment porter



Surveyor - monitor radiation, steam leaks, & gauges

"Robots for nuclear power plants", Taylor Moore, IAEA Bulletin, Autumn 1985 (31).

ROADMAP TEAM

EM's TDO sponsors development of this roadmap and assembled a Robotics Advisory Team consisting of:

- Rod Rimando, Chair DOE-EM TDO
- Paul Dixon, LANL
- Philip Heermann, SNL
- Tom Nance, SRNL
- Josh Mehling, NASA

This roadmap was developed by the DOE Robotics Roadmap Core Team, which consists of:

- Jason Wheeler, SNL
- Troy Harden, LANL
- John Lee, DOE-EM TDO
- Richard Minichan, SRNL
- Eric Kriikku, SRNL
- Wendell Chun, U Colorado, Denver
- Bill Hamel, U Tennessee
- Richard Voyles, Purdue

ROBOTICS DEVELOPMENT AND DEPLOYMENT STRATEGY

This roadmap provides recommendations based on a strategy with the following elements:

1. **Leverage technology and best practices.** Applicable technology is already available and being developed by:
   - DOE national laboratories
   - Universities
   - Industry
   - Other federal agencies & programs (e.g. National Robotics Initiative and the National Networks for Manufacturing Innovation program)
   - International entities
2. **Develop technology to address unique needs.**
   - The legacy waste clean-up mission presents unique challenges
   - Existing technology will be adapted for use where practical,
   - but some targeted development is anticipated

ROBOTICS DEVELOPMENT AND DEPLOYMENT STRATEGY (cont.)

3. **Prepare for future needs**
   - Follow a phased approach with near- to long-term perspectives
   - With many difficult challenges involved with multi-year technology development cycles
   - Technology development must begin ahead of time to ensure that solutions exist when needed
4. **Facilitate Technology pull**
   - Avoid developing technology that will sit on the shelf unused
     - Ensure awareness of the usefulness of the technology
     - Ensure awareness of the technology limitations that may make it impractical
   - Collaboration on:
     - Workforce educating technology developers on subtleties of the work
     - Developers educating the workforce on what the technology can do

ROBOTICS DEVELOPMENT AND DEPLOYMENT STRATEGY (cont.)

5. **Support site deployment**
   - The contracting mechanisms and workforce must be considered in evaluating the risks and rewards of technology insertion.
   - The contractor must be incentivized to deploy technology that can support and improve the DOE mission, even in the face of risks
   - Workforce will require training on new technology use & maintenance
6. **Transfer Technology**
   - Historically, DOE R&D has impacted many different domains
     - e.g. Argonne National Laboratory's Remote Control Division created the first robotic manipulators two decades before the introduction of industrial robots
   - Transferring the technology to industry can leverage economies of scale to reduce the cost of the technology to DOE-EM
   - Potential for the technology to increase U.S. workforce competitiveness

ROBOTICS DEVELOPMENT AND DEPLOYMENT STRATEGY (cont.)

7. **Facilitate International Common Interest Collaboration**
   - TDO will facilitate information sharing within worldwide chemical/radiological clean-up efforts
   - Benefit 1: leverage technology and best practices
   - Benefit 2: sharing information with international partners minimizes risk to U.S. interests

S.W.O.T. Analysis

What are the Internal/External Strengths and Weaknesses

Internal

Strengths

- Leverage ongoing work at LANL
- Ensure the contractor is incentivized to deploy technology that can support and improve the DOE mission, even in the face of risks

Weaknesses

- Fail to ensure facilitate technology pull
- Fail to ensure technology to address unique needs
- Fail to ensure collaboration between DOE workforce and technology developers
- Fail to ensure contracting mechanisms and workforce are considered in evaluating the risks and rewards of technology insertion

External

Opportunities

Leverage technology and best practices:

- DOE national laboratories & other federal agencies
- Domestic universities & industry
- International entities

Transferring the technology to industry:

- Leverage economies of scale to reduce cost to DOE
- Potential for the technology to increase U.S. workforce competitiveness

Threats

Fail to prepare for future needs:

- Large scope in storage or clean-up effort
- Decades of work & massive liability
- Requires a phased approach with near- to long-term perspectives
- Many difficult challenges involved with multi-year technology development cycles

Project Overview

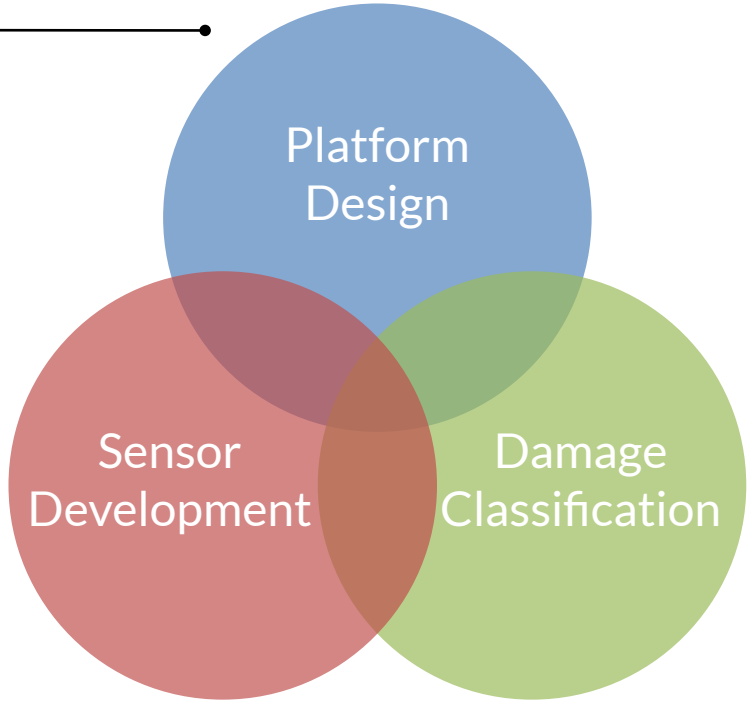
Three Primary Goals

Get There Safely

- COTS mobility & manipulation components
- SLAM
- Path / motion planning
- Robust safety systems

Get Good Data

- Always maintain as top priority
- Significantly lower probability of error compared to human



Make Good Decisions

- Longitudinal data
- Feature extraction
- Machine learning
- Databases

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Robotic Implementation Tasks

TASK NUMBER	TASK TITLE	FY MONTH											
		1	2	3	4	5	6	7	8	9	10	11	12
1	**Robot system development**												
1.1	Demonstrate robust, reliable, & safe path and motion planning												
1.1.1	Characterize the accuracy of spatial mapping												
1.1.2	Characterize the ability of the robot to execute path planning												
1.1.3	Characterize the ability of the robot to execute motion planning												
1.2	Characterize performance tradeoffs during data acquisition												
1.3	Explore machine learning methods for classification												
2	**Data aggregation & container health visualization**												
2.1	Complex (fused) feature definition for machine learning												
2.2	Combination of databases												
2.3	Exploration of visualization methodologies												
3	**Integration of scanning data with accountability inventory**												
3.1	Spatial Integration												
3.2	Content Integration												
4	**Facility deployment**												
4.1	Mockup testing at LANL												
4.2	Mockup testing at NMT												

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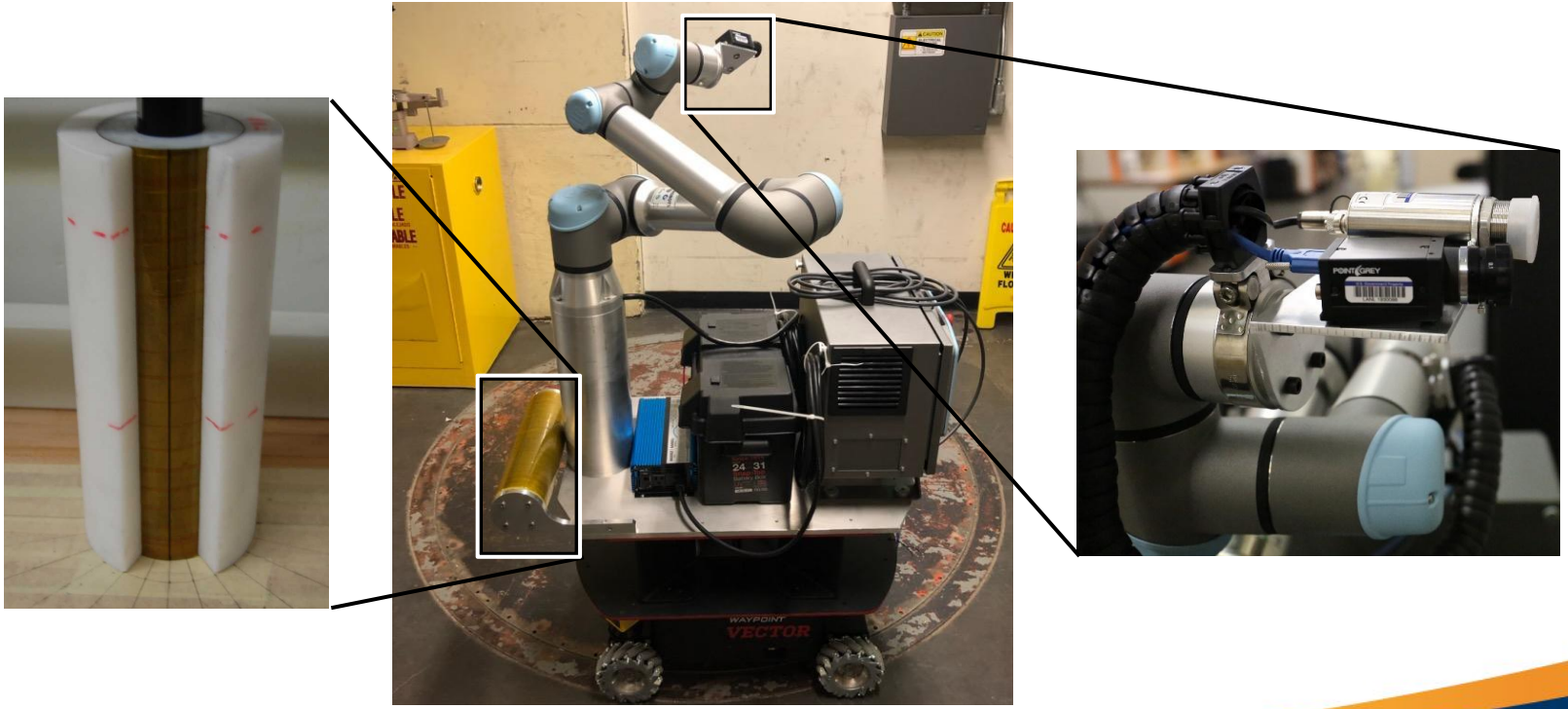
Test Platform



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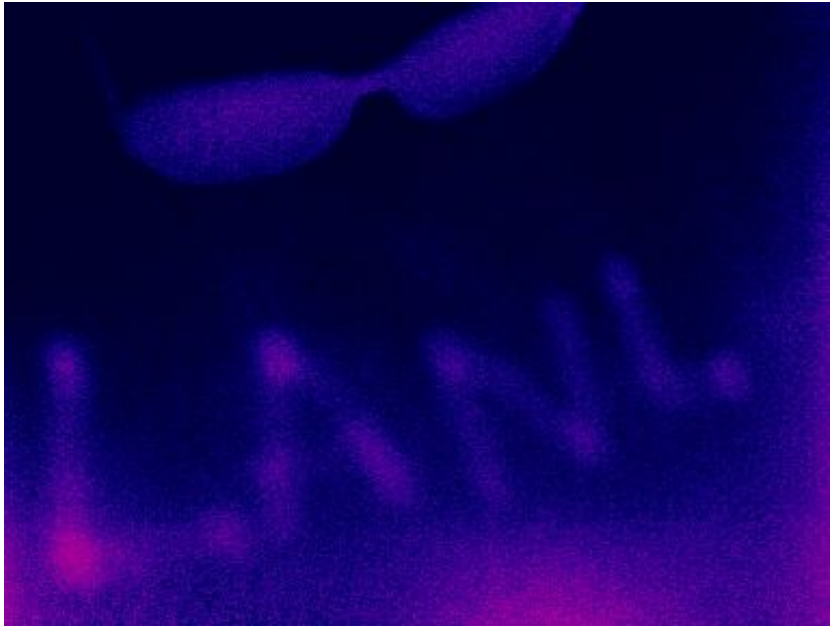
Test Environment

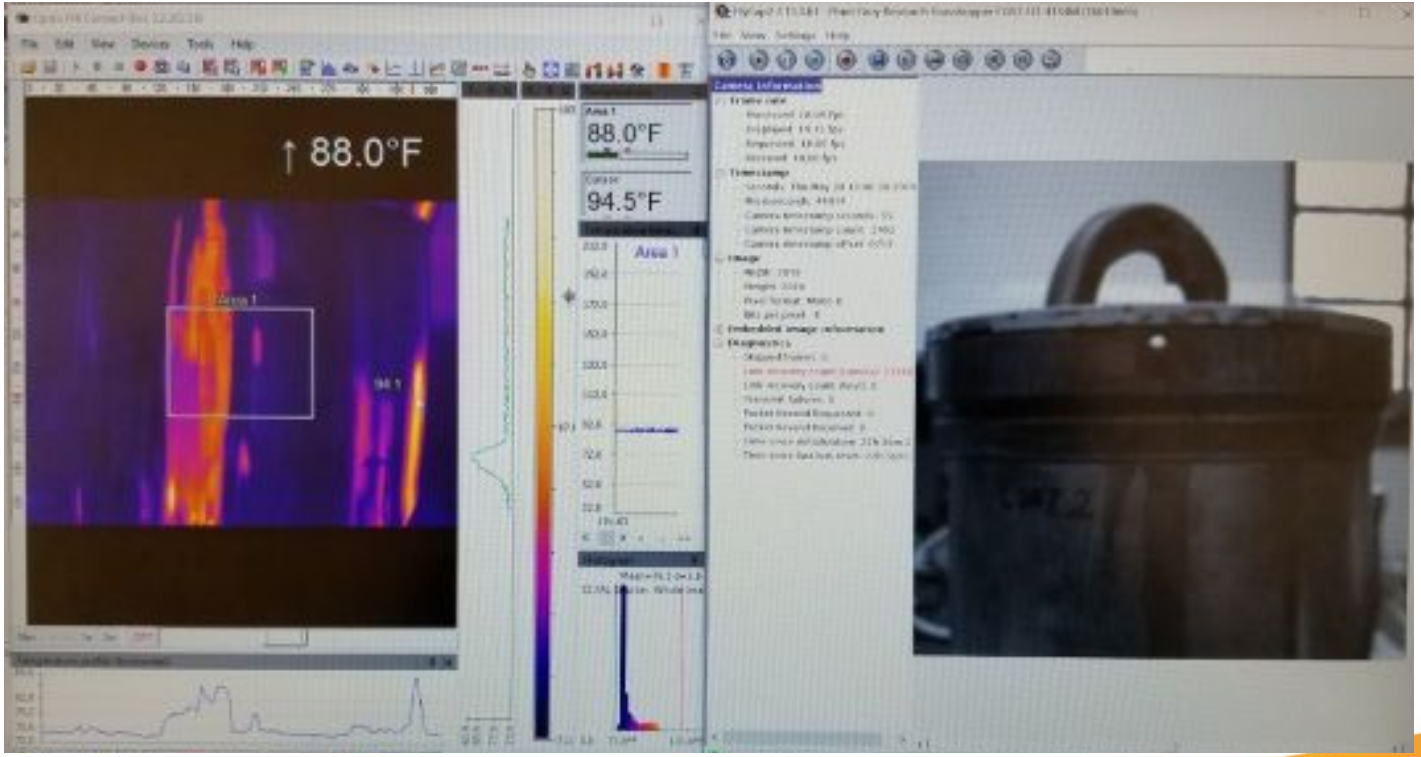
- Variety of container types and sizes
- Reviewing sealed source listings

Sensor Integration

- Working on communication protocols for the sensor peripherals
  - Optris Xi 400 Thermal Camera
  - FLIR Grasshopper 3 Visual Camera
  - Bridgeport Instruments Neutron Detector



Multi-Modal Imaging Example



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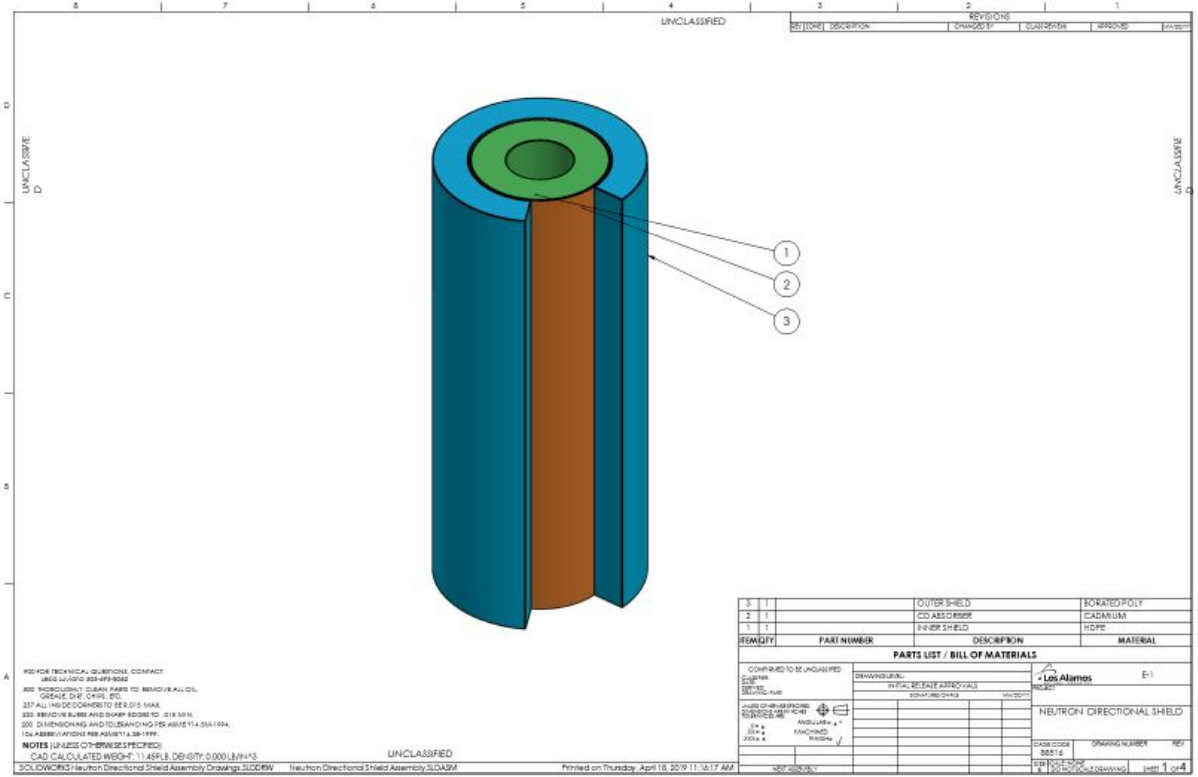
Preliminary Directional Detector Design:



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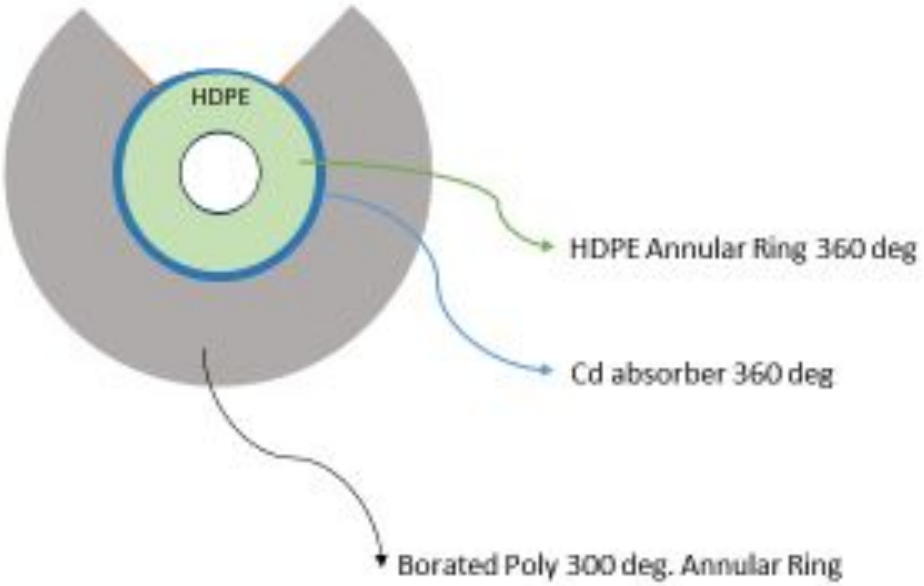

Conceptual Layout for a Directional Neutron Detector Shield

Test Bed Layout

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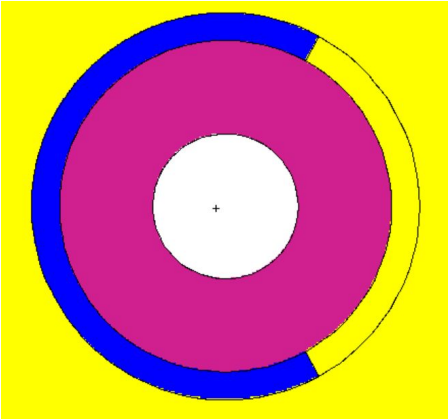
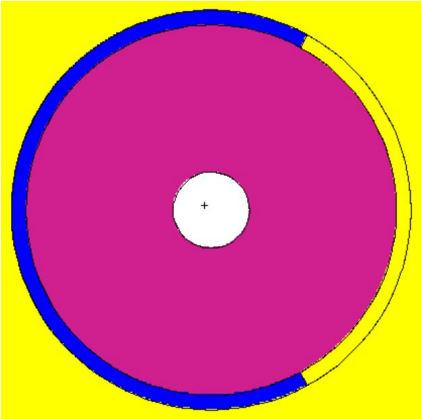
Preliminary Results:

Unmoderated		Abs Eff	Rel Eff
0 deg. Det CR	81.9	9.53%	100.00%
15	78.942	9.16%	96.11%
30	76.669	8.87%	93.13%
45	71.216	8.19%	85.96%
60	66.989	7.66%	80.41%
90	67.339	7.71%	80.87%
135	70.009	8.04%	84.37%
180	70.532	8.11%	85.06%
225	70.619	8.12%	85.18%
270	65.349	7.46%	78.25%
315	69.832	8.02%	84.14%
330	75.332	8.71%	91.37%
345	80.662	9.37%	98.37%
360	81.346	9.46%	99.27%

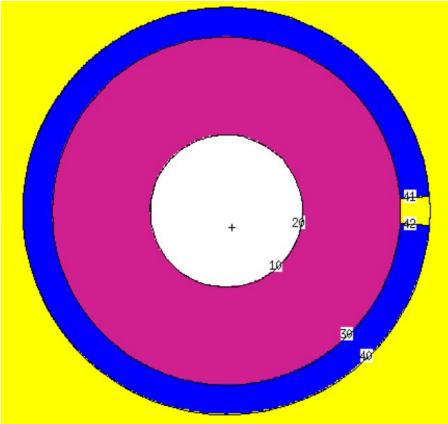
MCNP Optimization



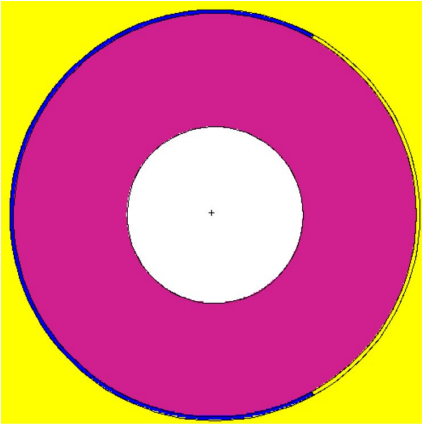
Base Case



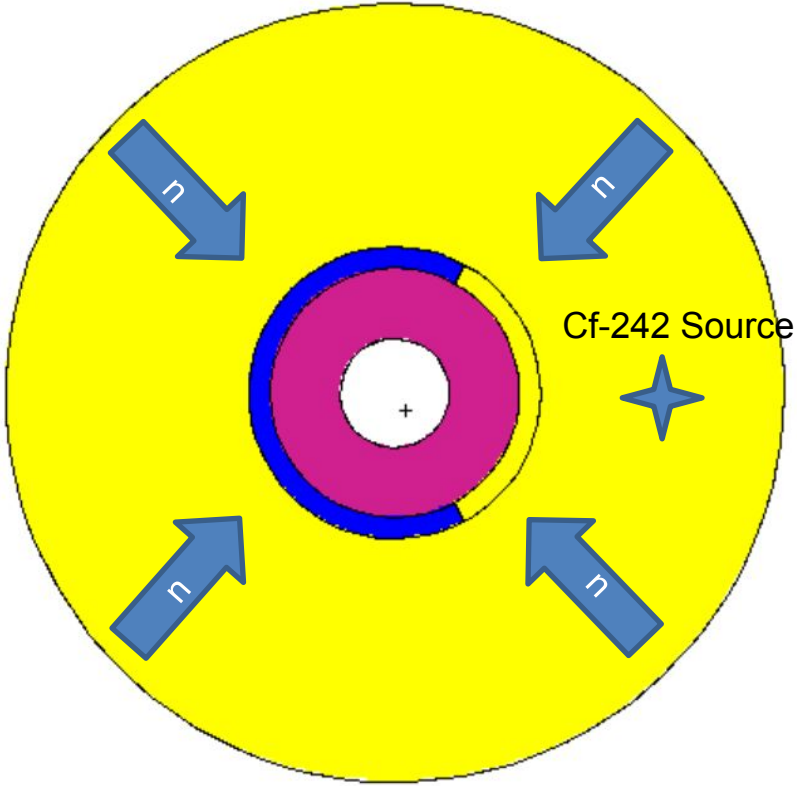
Variation of Poly



Variation of Window



Variation of Cadmium



Addition of Diffuse Neutron Background

Project Technical Areas

Divided by approach

Platform	Mobility	Manipulation	Sensing	Analytics
Physical Robustness	Mapping	Maximize View	Characterize Sensors	Optimize Accuracy
Power Management	Localization	Movement Efficiency	Physical Protection	Sensor Synergy
-	Path Planning	Motion Planning	Optimize Sampling	Quantify Accuracy
-	Collision Avoidance	Collision Avoidance		Quantify Perf. Ceiling

COTS

NMT

LANL

Future Effort

Other DOE Sites

Future Work

- Expand on preliminary SLAM success
- Explore damage detection sensitivity
- Explore feature extraction and machine learning approaches to classification
- Quantify dose reduction
- Address concerns related to wireless transmission, data security, robot contamination, etc.

Detector Testing and Characterization

- Determine Directional Sensitivity and Efficiency
- Refine MCNP model for detector design optimization
- Recommend Operational methods for Use (Motorized Rotation)
- Use Cf-252 as a surrogate for Pu-240
- Develop Facility Applications Based on Testing, Uncertainty Results and Operational Limitations

-end-

[backup slides]

Scope of Work

Pilot 1

1. Select and acquire two manipulators capable of rapid integration with a wide range of mobility platforms.
2. Select and acquire camera systems for integration with robot manipulator.
3. Integrate hardware and control software for mobility base, manipulator, camera systems, and radiation detectors
4. Conduct proof of concept testing to quantify container scan time, container scan percentage, and container scan accuracy.
5. Collect and analyze all data aggregated to evaluate container health in current form and future state. A major component of the proof of concept is to determine if the robot can collect the necessary data for container health monitoring.

Key Performance Metrics

Pilot 1

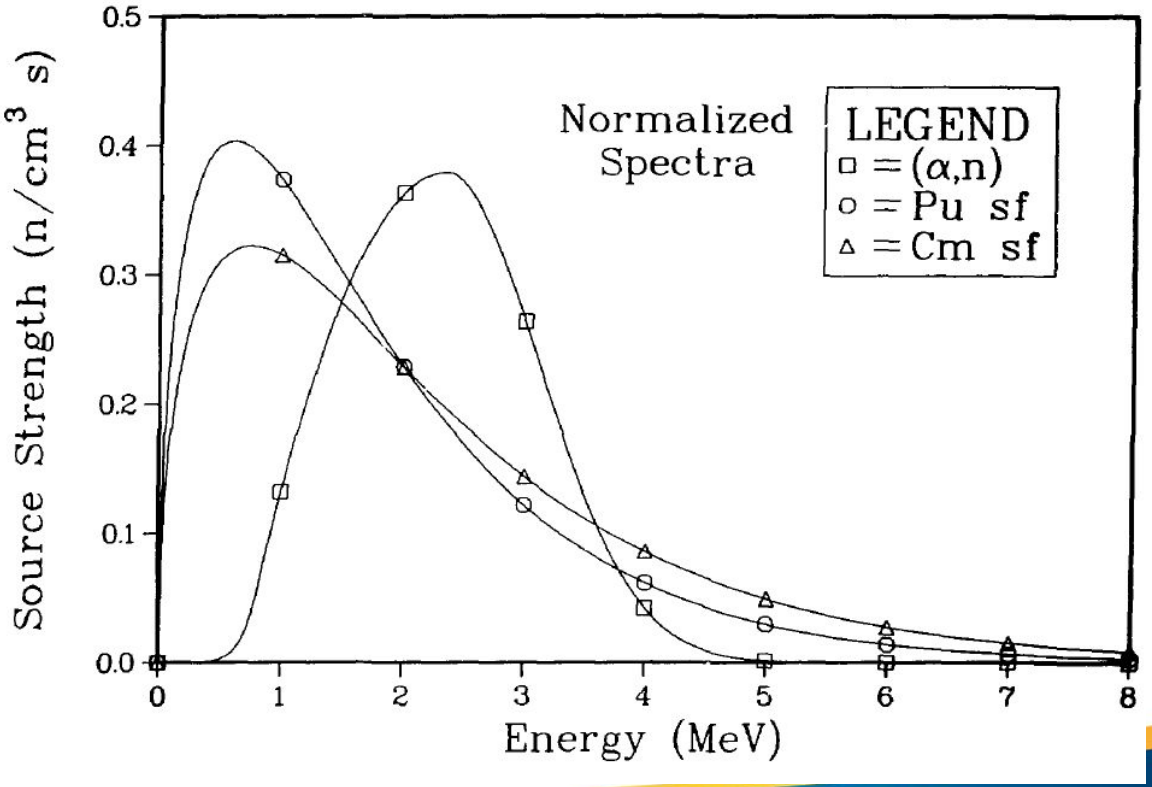
1. Cycle time - Measure the time that it takes for the robot to complete one sequence. It will be used to compare the robot's efficiency of operation with the previous manual/sporadic container measurement because of the high dose exposure.
2. Cycles complete and containers measured - Measure the number of sequences completed with the robot and the number of containers that are measured during a cycle. In many cases, it can indicate how many operations have been successful.
3. Dose Reduction – Quantify the worker dose savings compared to container evaluations for PF-4 vault retrieved containers. Quantify the equivalent worker dose that would be received with increased container surveys matching the efficiency of robotic system.

Successful Proof-of-Concept Outcomes

Pilot 1

1. Establish baseline functionality of mimicking container inspection a human would do with a robot to reduce worker dose.
2. Quantify what the robot is able to do beyond a person's capability without taking dose.
3. Robots ability to navigate designated path.
4. Autonomous and/or semi-autonomous navigation to location avoiding both known and unknown obstacles and the ability to identify issues with little to no human intervention.
5. Visual inspection beyond the human eye including bulging, dents, scratches, and other damages.
6. Higher amount of inspection through radiation detection (gamma and/or neutron and thermal) and collection of this data over time.
7. Visualization of changes to containers overtime through robotic inspections including bulging, dents, scratches, and other damages often overlooked by personnel.
8. Database of aggregated data that can maintain historical data that shows change over a period of time to ultimately determine the health of the container.
9. Robot overall movement is accurate, repeatable, and predictable.

Spontaneous Fission Vs (alpha,n) Spectra



Detector Operation

Neutron Detection

New technology based on $^{10}B/ZnS(Ag)$

A new and innovative type of thermal neutron detection technology uses scintillating, neutron-sensitive films that can be applied to substrates of various shapes.

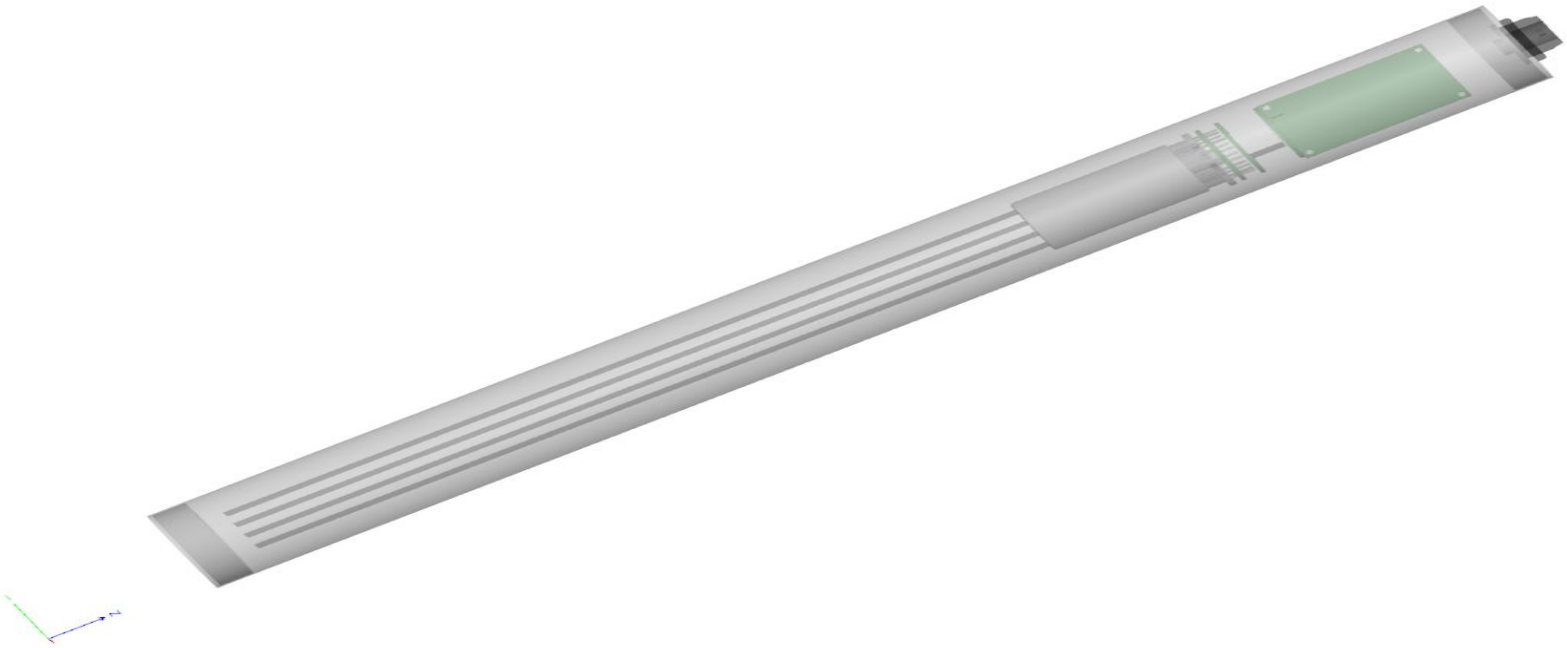
Standard round detectors may replace pressurized tubes, but many other forms, such as bars and paddles, will soon be available as well.

Our technology supports phoswiches, which makes it possible to combine neutron detection and gamma-ray measurement into a single channel of electronics.

We have formulated an environmentally friendly neutron-detecting compound, which minimizes disposal cost.

- **Shapes and forms**
  - Cylindrical
  - Square & hexagonal profiles
  - Bars and paddles
- **Safe**
  - No exposed high voltage
  - No hazardous substances
  - No pressurized tubes
  - No disposal problem
- **Easy to use**
  - Power-up and count
  - Can read data via USB

Digital ^{10}B/ZnS(Ag) Detector Design



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Preliminary Results:

Preliminary Bridgeport Neutron Detector Source Calcs

Isotope Products		
Source	A7-869	
DOB	11/1/2002	
Activity	1660	uCi
Flux	7.125E+06	n/sec
Uncertainity	5.80%	3 sigma
T 1/2	2.645	yrs

Decay Calculations

Date of interest	5/3/2019	
Elapsed Time	6027	days
Activity of Interest	21.98	uCi
IP Flux on Date	94081	n/sec
Panda Flux	96730	n/sec

Panda Manual Data		
T 1/2	2.646	hrs
neutron Yield	2.34E+12	n/sec/g
	4.40E+09	n/srec/Ci
Spec Act	558	Ci/g
Neu E ave	2.14	MeV

Fluence Calculations		
Distance to Detector CL	15	inches
4 π area	18241.47	cm^2
Detector Area	154.84	cm^2
Fluence	5.16	$n/cm^2/sec$
Detector Incidence	798.58	n/sec

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- Founded in 1889, STEM focused
- Enrollment: 2,000
- NSF study of Baccalaureate Origins of S&E Doctorate Recipients US ranked New Mexico Tech as 15th in the nation (#1 ranked public institution)
- Mechanical Eng. program grown rapidly: roughly ¼ of all students, BS, MS, ME, PhD
- EMRTC: 40 mi^2 field laboratory; more than 30 test sites, gun ranges, other research facilities; ability to conduct tests involving over 20,000 pounds of explosives
- **Takeaway**: good engineering students, unique facilities, close to LANL.

Initial Engineering Evaluations of the New In-glovebox Container Designs

Jude M. Oka, Tim A. Stone, Tristan Karns, Paul H. Smith, Young Tao, Murray Moore

Los Alamos NATIONAL LABORATORY EST. 1943

Managed by Triad National Security, LLC for the U.S. Department of Energy's NNSA

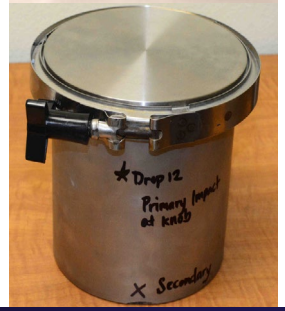
LA-UR-19-26081

Prototypes for testing

Strike-less ROCK

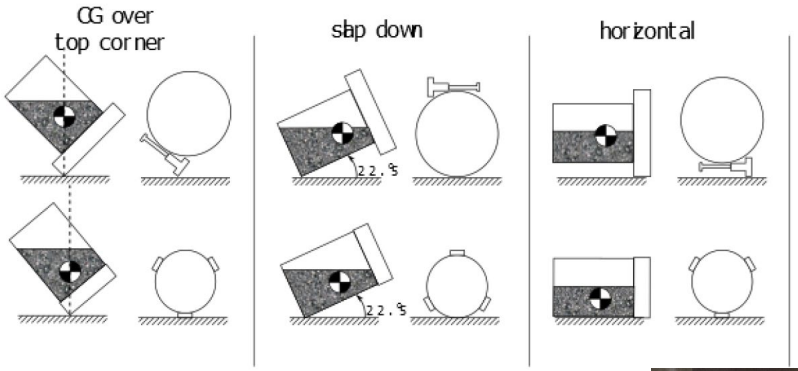
Threaded ROCK

K-Flange



Criticality requirements crediting

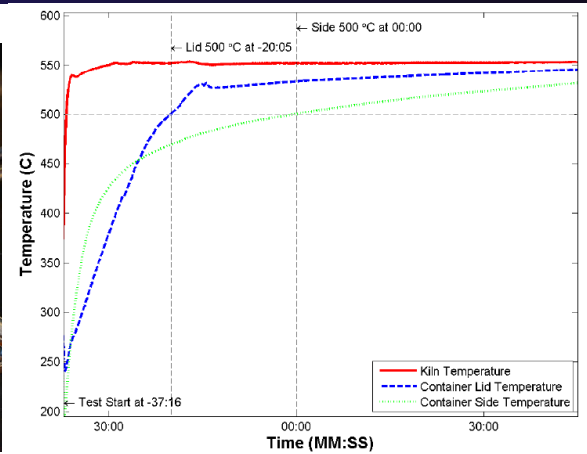
Accidental drop

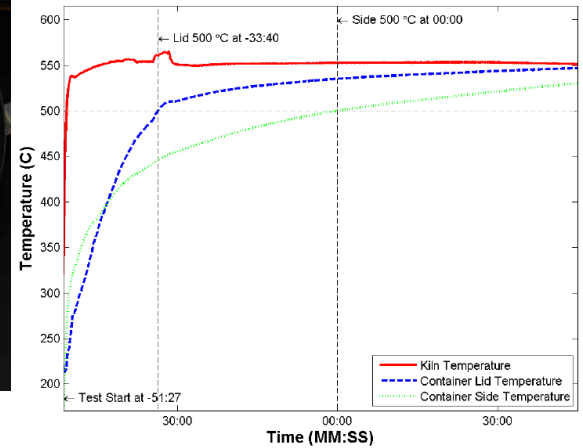


9.0kg gross weight for each container

Postulated in-glovebox fire

Criticality requirements testing - NMTech

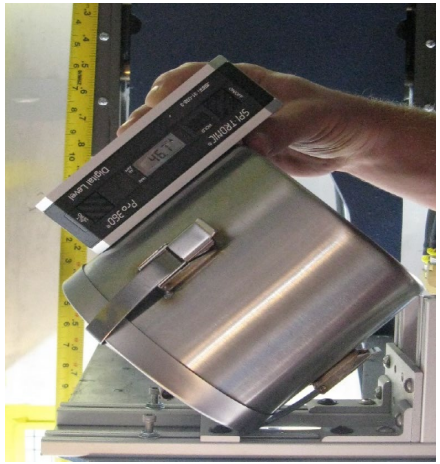




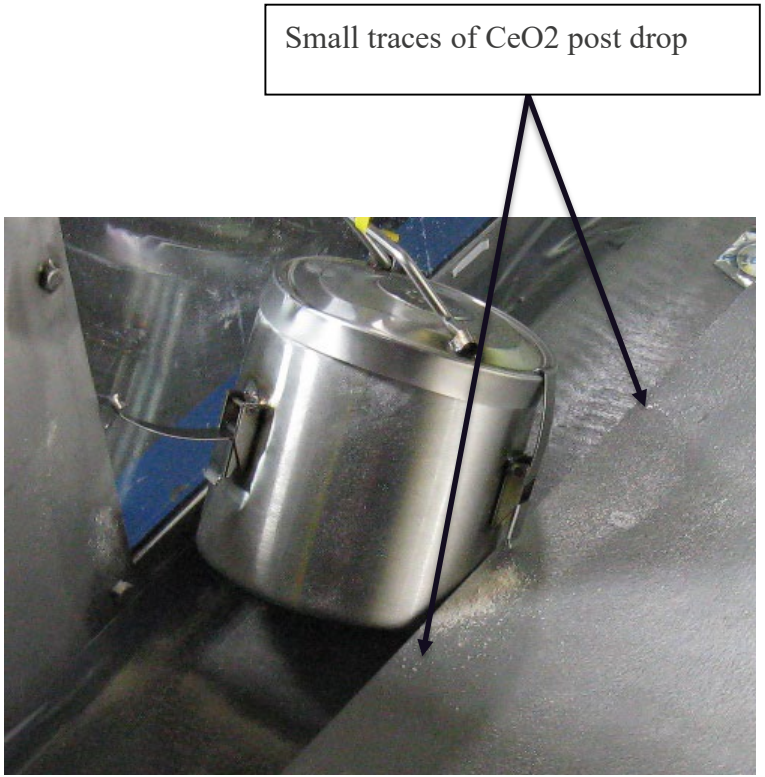
NMTech Results

Container Type	Drop Orient	Post Drop Water Ingress (g)	Post Fire Water Ingress (g)
K Flange	Horizontal	2.7	35.6
K Flange	Slap	2.9	93.8
K Flange	COMTC	3.6	85.9
ROCK	COMBC	1.7	44.1
ROCK	Horizontal	36.2	84.6
ROCK	Slap	43.3	218.8
ROCK	COMTC	1,369.6	407.3

RRFMC Testing of the Latch ROCK



RRFMC Testing of the Latch ROCK



Drop test results

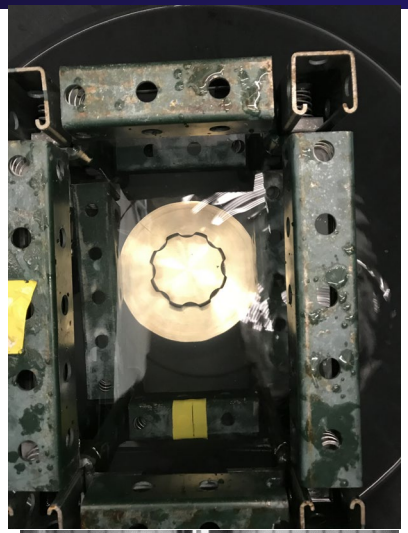
CeO2 MAR (g)	401.1
Gross Weight Pre-Drop (g)	9001.5
Gross Weight Post-Drop (g)	9001.6
Released Mass, dm (g)	1.8
Drop Orientation	CG over top corner
Pre-Drop Angle	46.7°
Drop Height (ft)	12
Drop Energy (joules)	324
Post drop water ingress (g)	N/A
DR value	4.5E-03

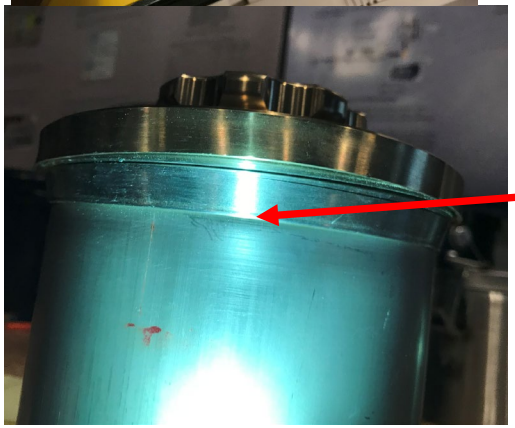
RRFMC results

	Respirable mass, g	Airborne mass, g
RRFMC Background at 324 J	6.02E-05 ± 5.41E-05	9.11E-05 ± 8.39E-05
Net Latched design	1.94E-02 ± 1.31E-03	3.22E-02 ± 2.12E-03

RRFMC Testing of the threaded ROCK

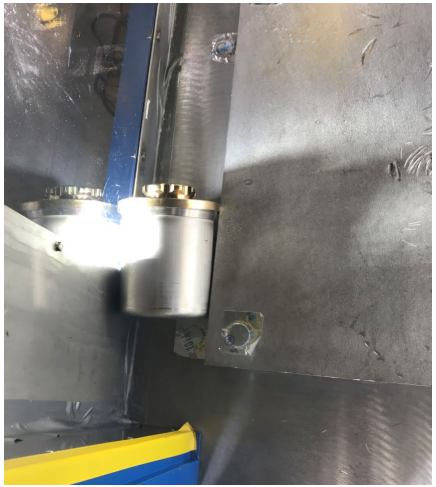
Pre drop
Water ingress test :
18 mil



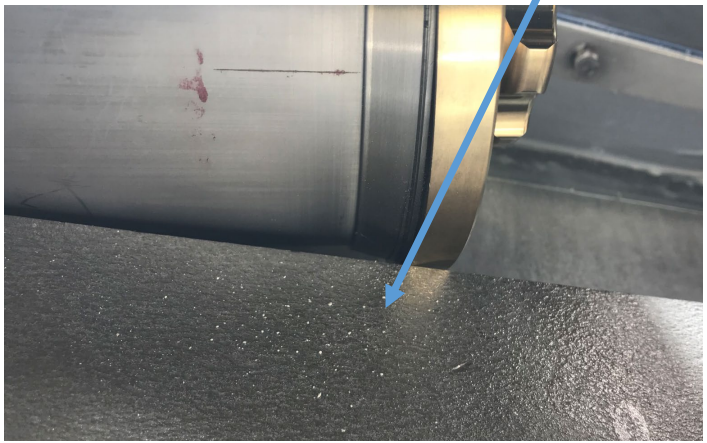


Post drop
deformation

RRFMC Testing of the threaded ROCK



Very small amounts of CeO2



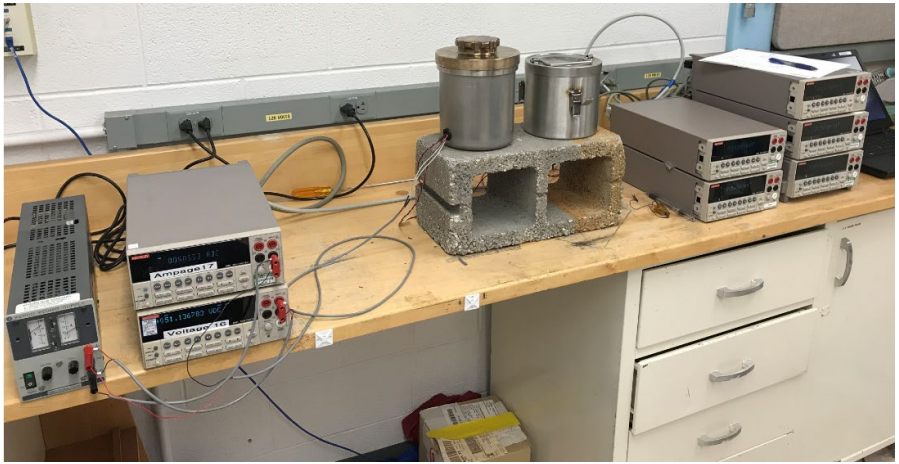
Drop test results

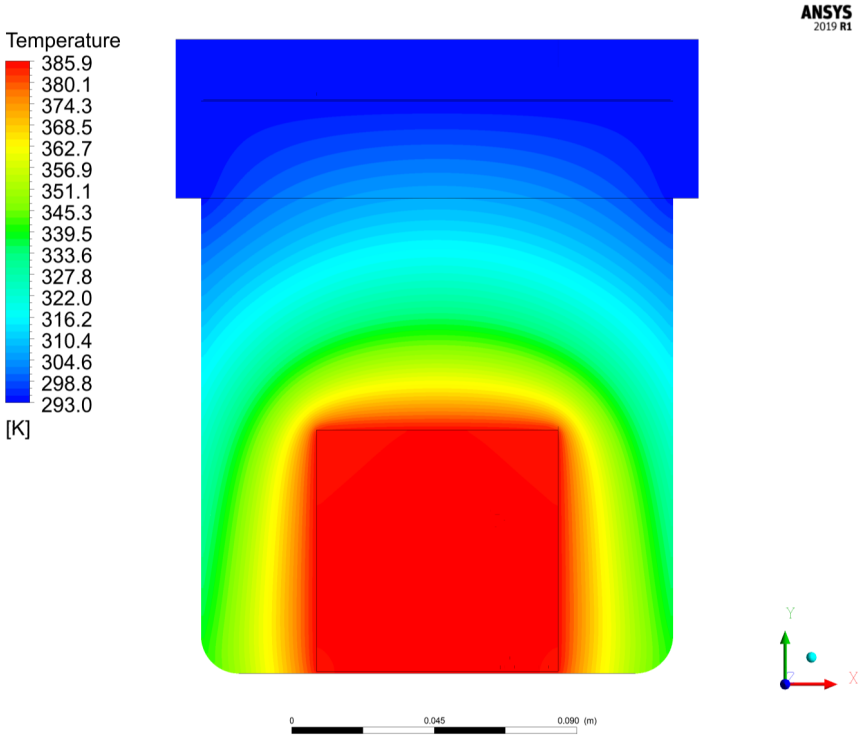
CeO2 MAR (g)	402.5
Gross Weight Pre-Drop (g)	9003.7
Gross Weight Post-Drop (g)	9003.7
Released Mass, dm (g)	0.0872
Drop Orientation	CG over top corner
Pre-Drop Angle	41.5°
Drop Height (ft)	12
Drop Energy (joules)	324
Post drop water ingress (g)	41.1
DR value	2.17E-04

RRFMC results

	Respirable mass, g	Airborne mass, g
RRFMC Background at 324 J	6.02E-05 ± 5.41E-05	9.11E-05 ± 8.39E-05
Net Threaded design	5.59E-04 ± 6.81E-05	8.90E-04 ± 1.10E-04

Heat loading and CFD modeling





Questions

TURN TO CLOSE BEFORE LIFTING!
CLOSED
OPEN
031105073B
5QT
031105073B

RRFMC – Respirable Release Fraction Measurement Chamber

Aug. 7, 2019

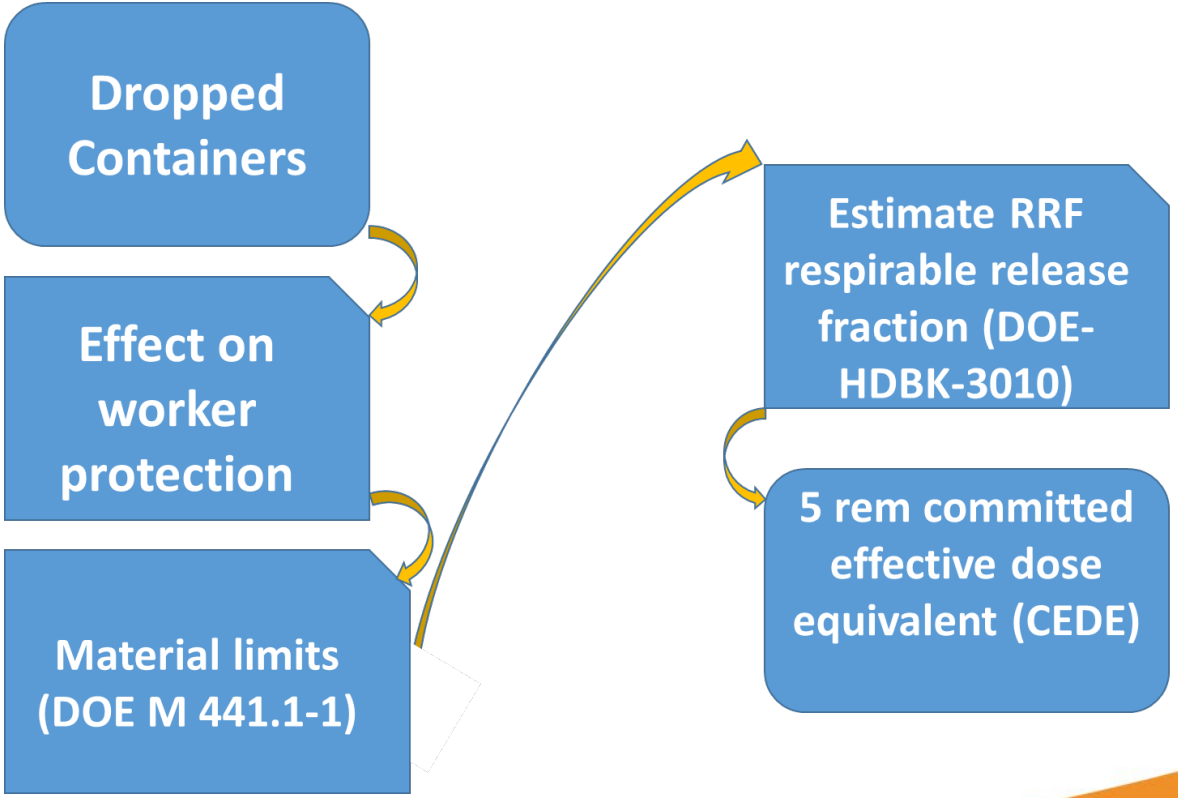
LA-UR-19-27692

Yong Tao, Murray E. Moore, Paul H. Smith, Elizabeth Kelly, Tristan Karns, Kirk P. Reeves Tim A. Stone, Jude M. Oka

UNCLASSIFIED

Introduction

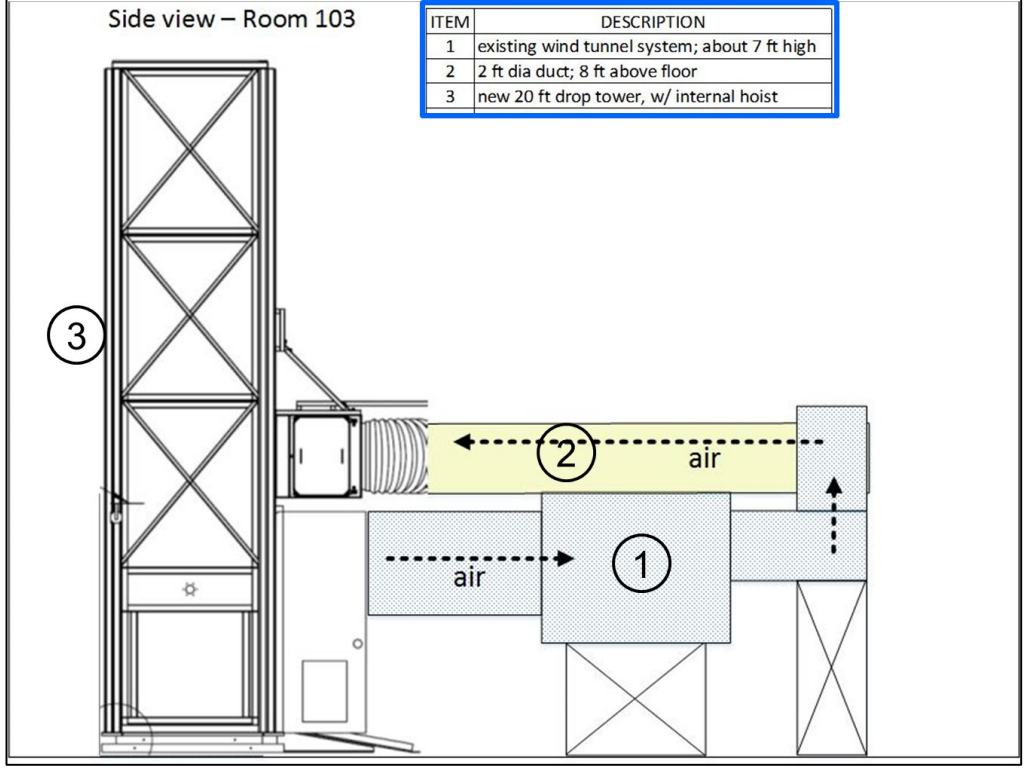
- Nuclear material storage container (NMSC)
- Nuclear material package manual DOE M 441.1-1
- Respirable Release Fraction Measurement Chamber (RRFMC)
- Purposes of this study



Methods

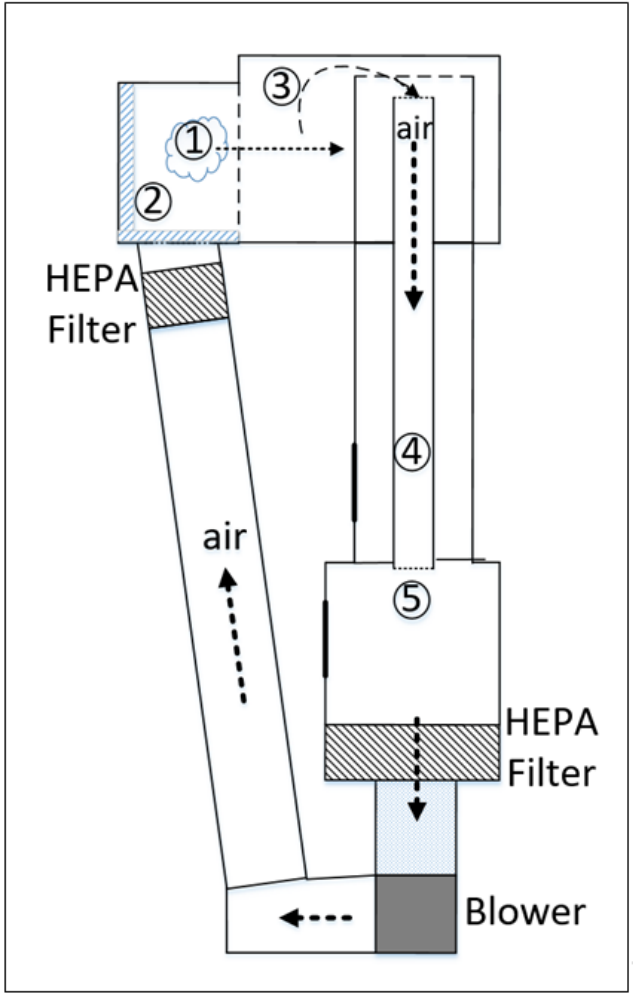
RRFMC

- Measure ARF and RRF of drop tested containers.
- 6.10 m tall, 1.42 m×1.02 m inside footprint, HEPA filtered, airtight drop tower.
- Remote controlled Lansmont PDT 80 drop tester.
- Test up to 68.0 kg mass and 4.88 m height.
- Non-toxic cerium oxide analytical test powder.
- Aerodynamic Particle Sizer (APS) measures aerosol concentration and size.



- Dual axis hi-speed videography.
- Meets NQA-1 subpart 2.4 for R&D work.

(a)



(b)

(1)Drop impact location

(2)Plenum to reroute air inside RRFMC

(3) Air mixer

(4) Duct with air velocity probe

(5) Aerosol measurement location

Test Procedure

- Clean tower & prep test container.
- Load container at desired impact orientation.
- Set up hi-speed video (optional).
- Close RRFMC door. HEPA filter the air.
- Monitor aerosol concentration.
- Drop the container after background aerosol criterion is reached.

- Kinetic impact increases airborne concentration. Monitor until background concentration is re-attained.
- Generic mixing system is suitable for single point air sampling.
- Aerosol transmission ratio and effective density were measured in previous work.

Calculation methods

Respirable mass (AED < 10 μm) of released aerosol (*mr, g*):

$$mr = QR \times Ts \times \sum_{i=1}^{t} \left[\sum_{j=1}^{D_a=10} \left(\frac{C_{ij}}{P_j} \right) \right],$$

C_{ij} = aerosol mass concentration (g/m^3) at interval (*i*) in sampling bin (*j*),

P_j = transmission ratio for particles in the APS sampling bin (*j*),

QR = RRFMC total air flow rate (*m^3/min*),

Ts = APS sampling interval (1 min).

- Airborne mass (AED < 20 μm) of released aerosol (*ma, g*) has the same format with different limits.

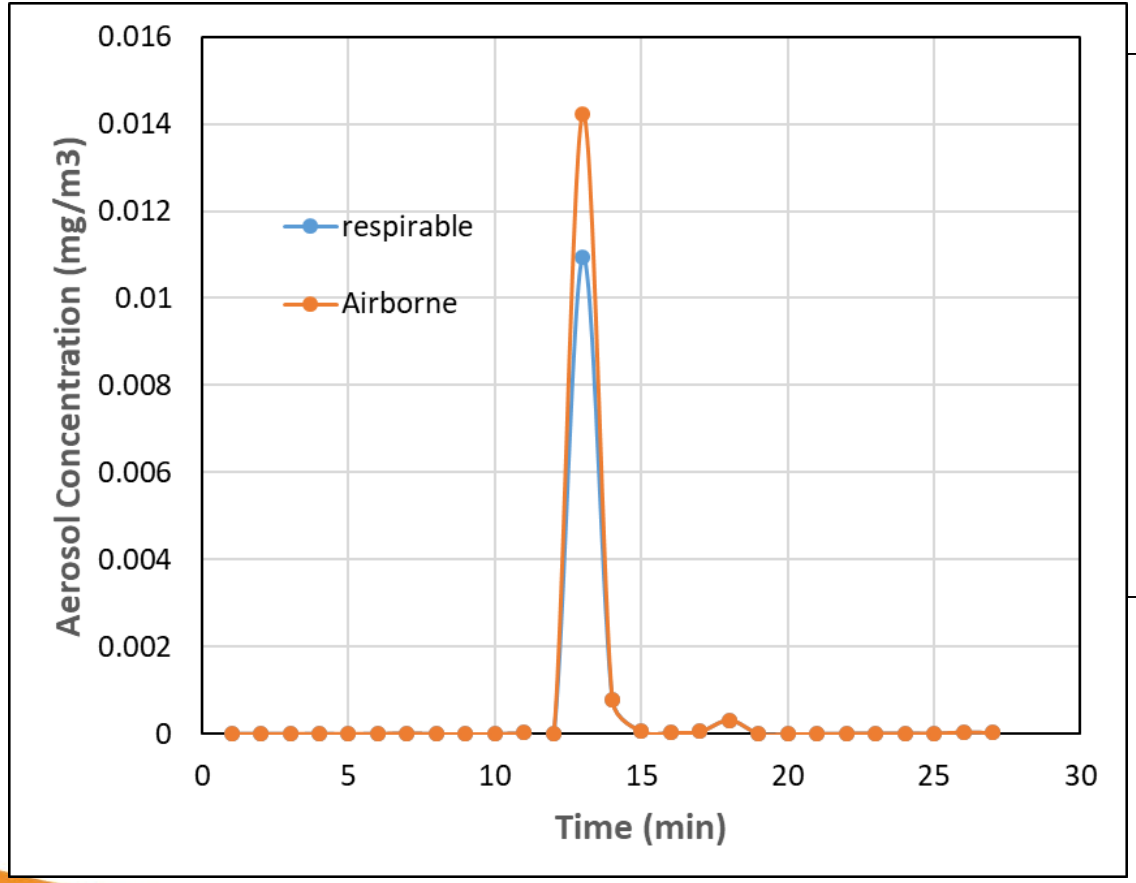
- Five-component equation:

*Source Term(S) = MAR*DR*ARF*RF*LPF*

- Respirable Release Fraction

$$RRF = DR \cdot ARF \cdot RF = \frac{mr}{MAR}$$

Aerosol Background for Control (Clean) Impacts



netmr = mr – Bkg

netmr = net respirable mass

mr = measured respirable mass

Bkg = average background of aerosol for clean impacts at the same drop potential energy

Aerosol concentration spike measured for 324 J clean control drop

Spilled Uptake Mass (SUM) and Spilled Uptake Factor (SUF)

- The SUM estimates worker inhalation of respirable particles from a dropped container.

$$SUM = Q \cdot \frac{netmr}{A \cdot V} \cdot (1 - \exp(-At))$$

netmr = net released respirable mass (g),

Q = standard person breathing rate (1.2 m^3 h^{-1}),

A = ventilation rate of the room, selected for LANL operational reasons, (8.0 h^{-1})

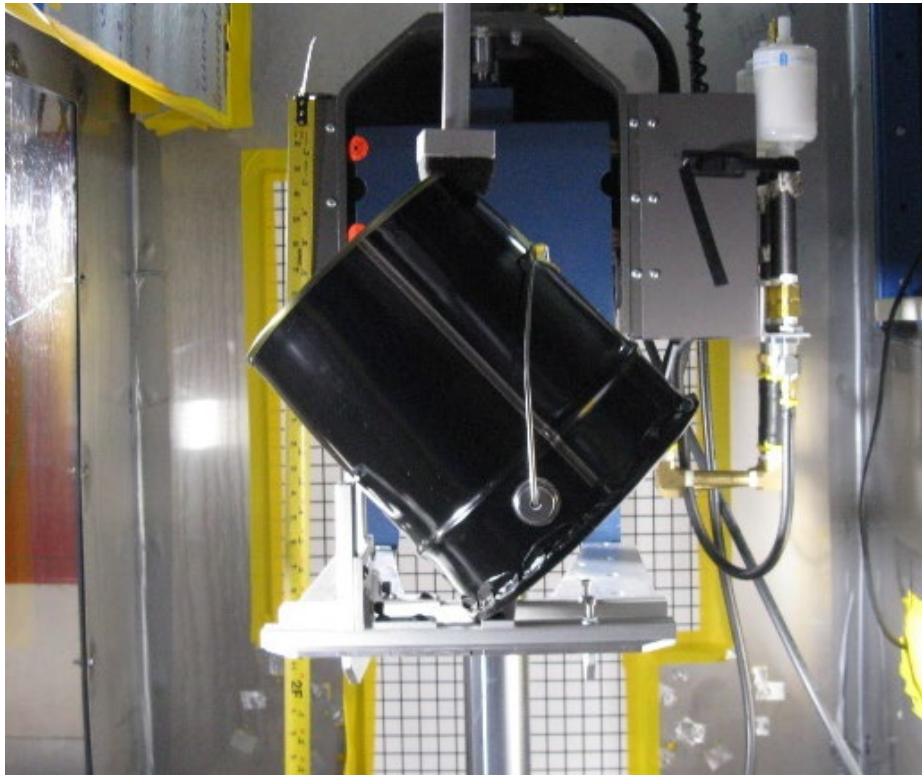
V = room volume (70.8 m^3), selected for LANL operational reasons, and,

t = time duration for a person in the room after the container is dropped (0.167 h) (according to DOE M 441.1-1).

- SUF = SUM / (spilled mass)

Drop Test Conditions

- 2 Gal pail test containers.
- 8.7 kg steel cylinder pay load.
- Dried CeO_2 test powder (3.4 to 5.5 g)
- Total gross weight about 9.46 kg
- Drop height: 4.78 m
- Drop orientation:
  - Test 1-3, 5, 6: with lid, CG over top corner.
  - Test 4: without lid, CG over bottom corner.



Test Results

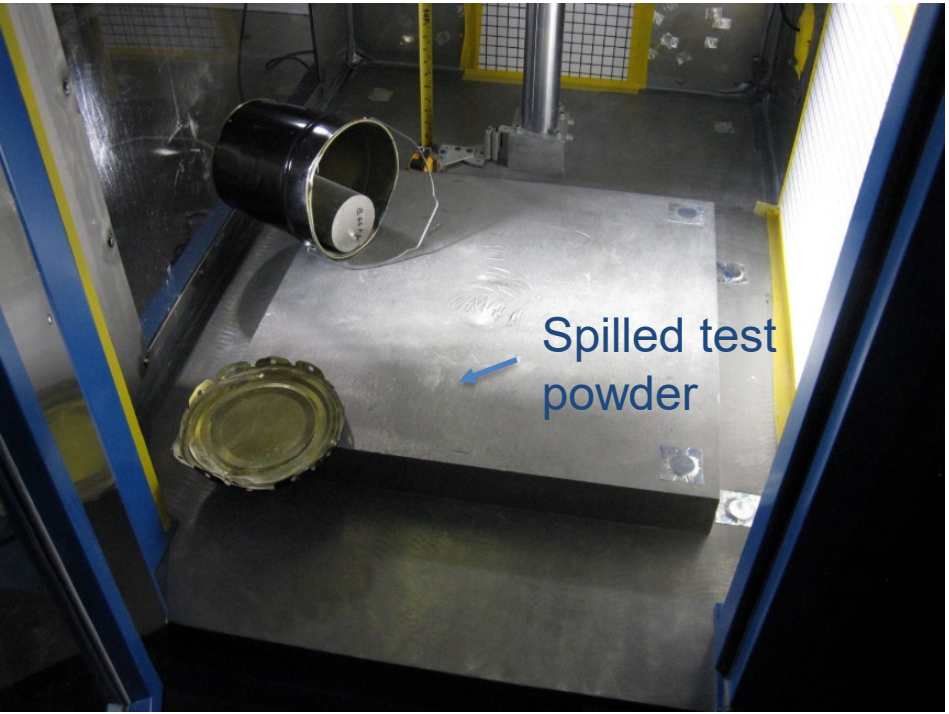
Table 1. Summary of drop test conditions and test results

	Drop Height, m	Gross Mass, g	Test powder MAR, g	Spilled mass, g	RRF	SUM, g	SUF
Test 1	4.78	9459.9	3.4	2.3	8.74E-03	4.64E-05	2.02E-05
Test 2[1]	4.78	9455.1	5.1	0.2	2.61E-03	2.07E-05	1.04E-04
Test 3	4.78	9459.2	4.1	2.8	1.86E-02	1.19E-04	4.25E-05
Test 4[2]	4.78	9310.9	4.6	1.3	2.88E-03	2.07E-05	1.59E-05
Test 5	4.78	9471.3	4.7	1.8	5.52E-03	4.04E-05	2.25E-05
Test 6	4.78	9472.1	5.5	2.7	9.18E-03	7.88E-05	2.92E-05

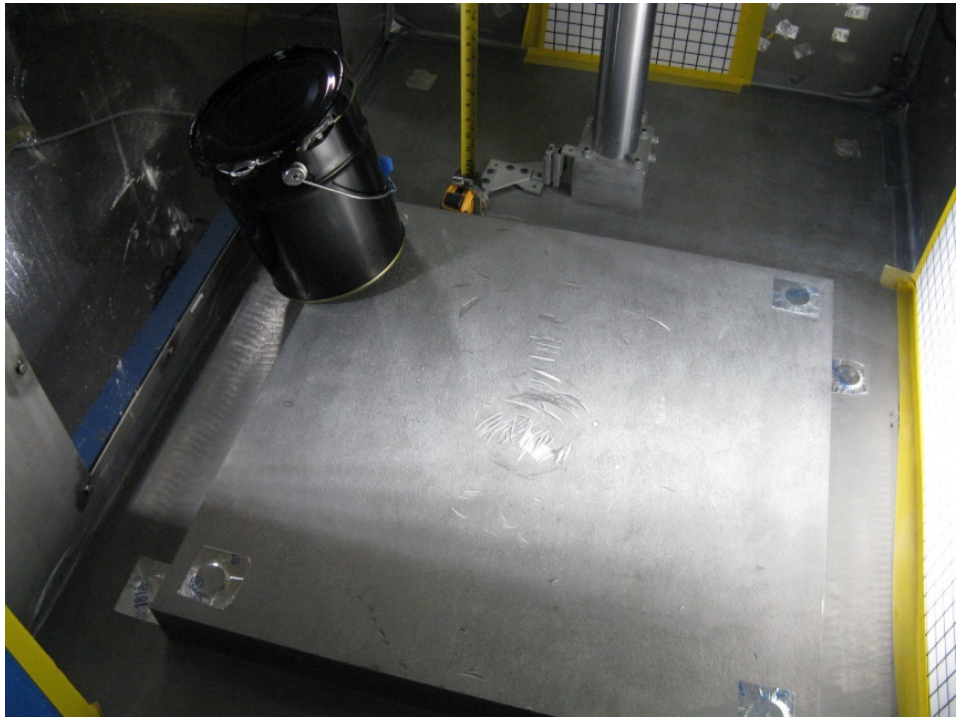
1. **Lid stayed on after drop (Figure 5b).**
2. **Drop tested without lid, CG over bottom corner orientation.**

Spilled mass is the difference between the initial and post drop mass of the tested pail.

Positions of the container after dropping



Test 1



Test 2

Experimental set up comparison to Gao et al. 2013 (Y12)

	LANL	Y12
Test Powder	CeO_2, 3.4 – 5.5 g	WO_3, 1936 g
Drop Potential Energy	445 J	25 J and 50 J
Test Equipment	RRFMC	Full-scale environmental chamber

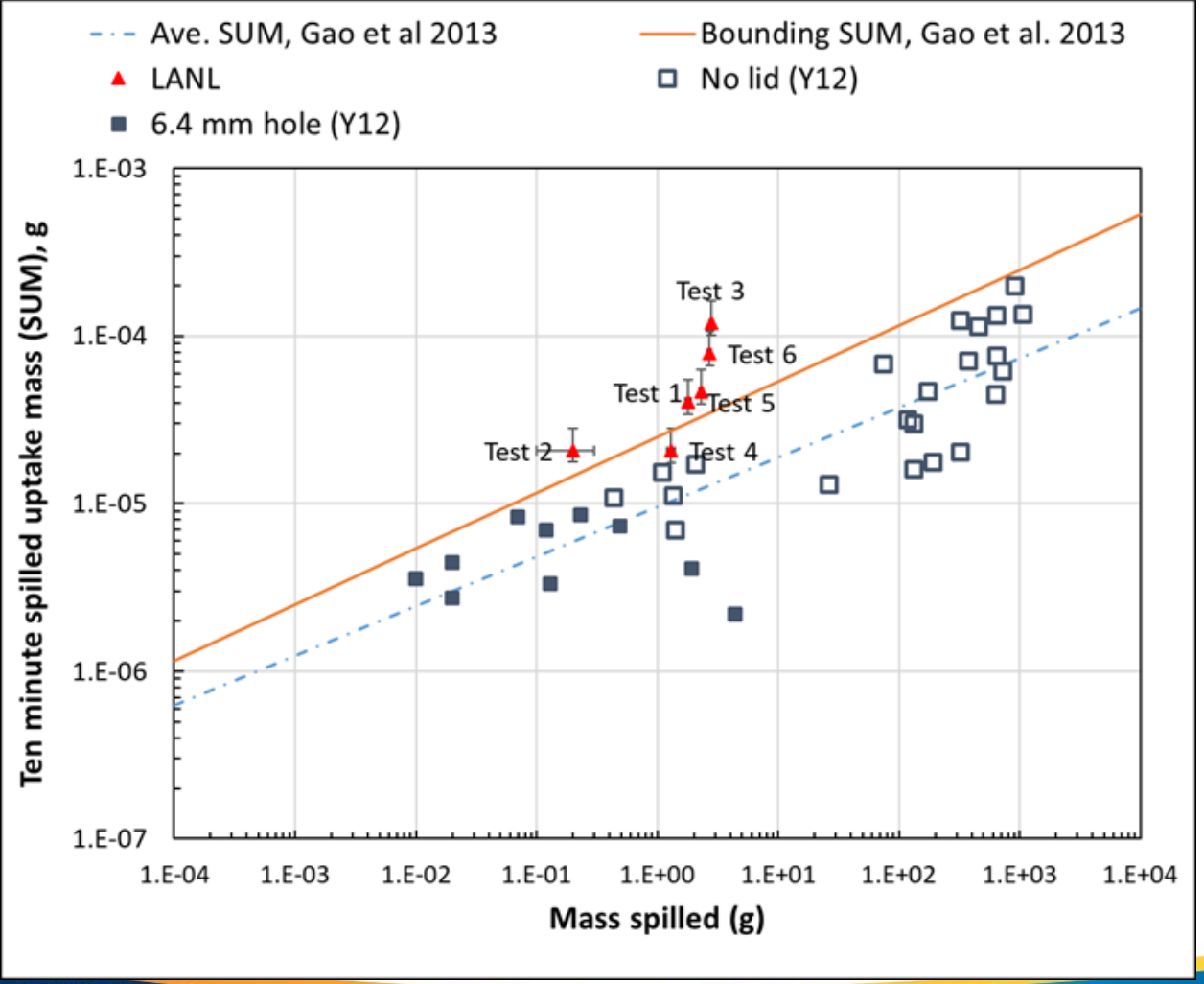
LANL

FIGURE 2. Container with powder (without lid, or covered by a lid with a hole)

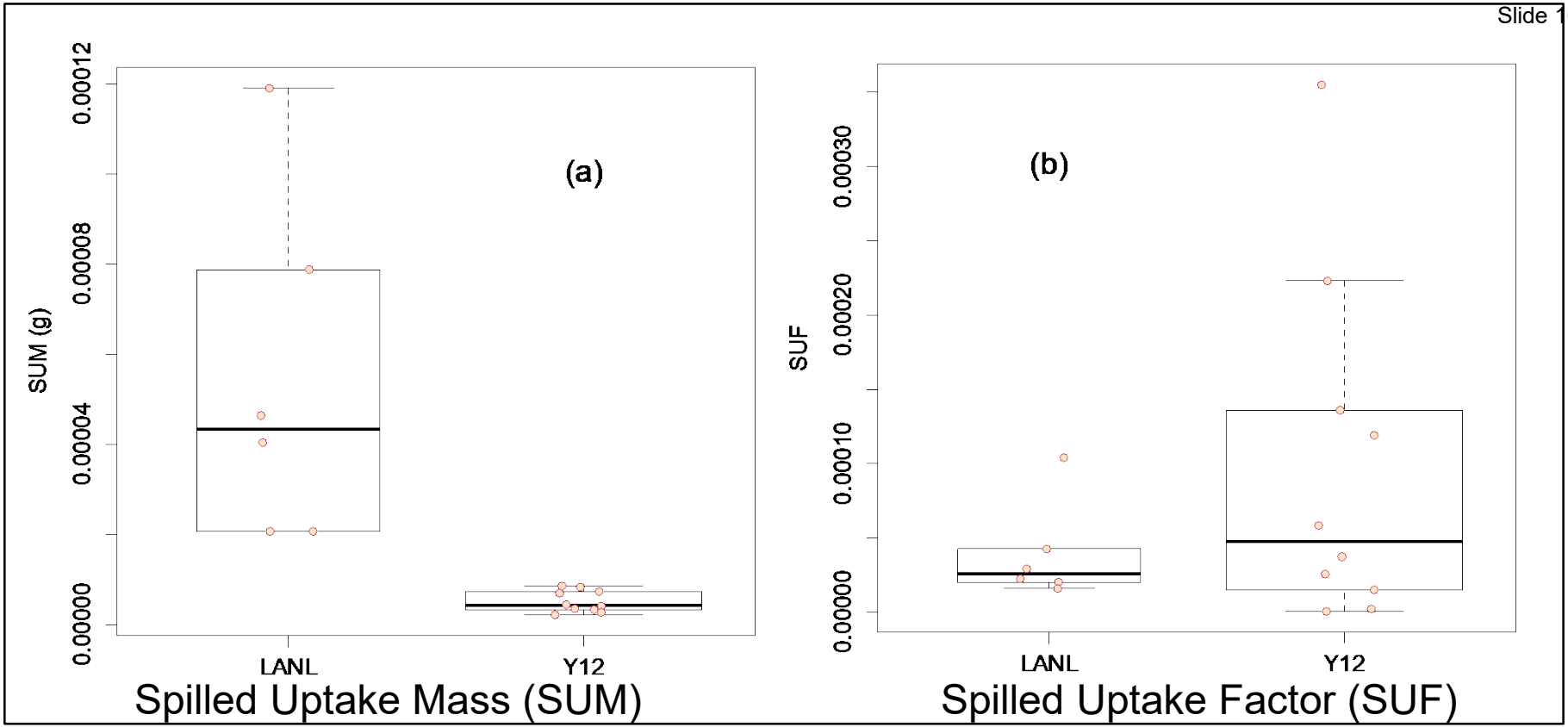
Y12

UNCLASSIFIED

SUM comparison to Gao et al. 2013 (Y12)



- Considerable variability in both LANL and Y12 measurements
- LANL measurements have higher SUM



Comparison to Y12 results that covered by a lid with 6.4 mm hole

Discussion and conclusions

- The RRFMC measures released powder during the dynamic phase of an impact event.
- To verify compliance with DOE M441.1-1, multiple drops of the container should be considered, due to test result variabilities.
- The RRFMC is operational and addition applications are being considered.

Selected references

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- US DOE handbook DOE-HDBK-3010-94. Airborne release fractions/rates and respirable fractions for nonreactor nuclear facilities. US Department of Energy, 1994

QUESTIONS?

THANK
YOU

Lawrence Livermore National Laboratory
Nuclear Material Packaging Site Update

NMP Program Review

Andrew Wysong
Nuclear Operations Integration Manager

August 20, 2019

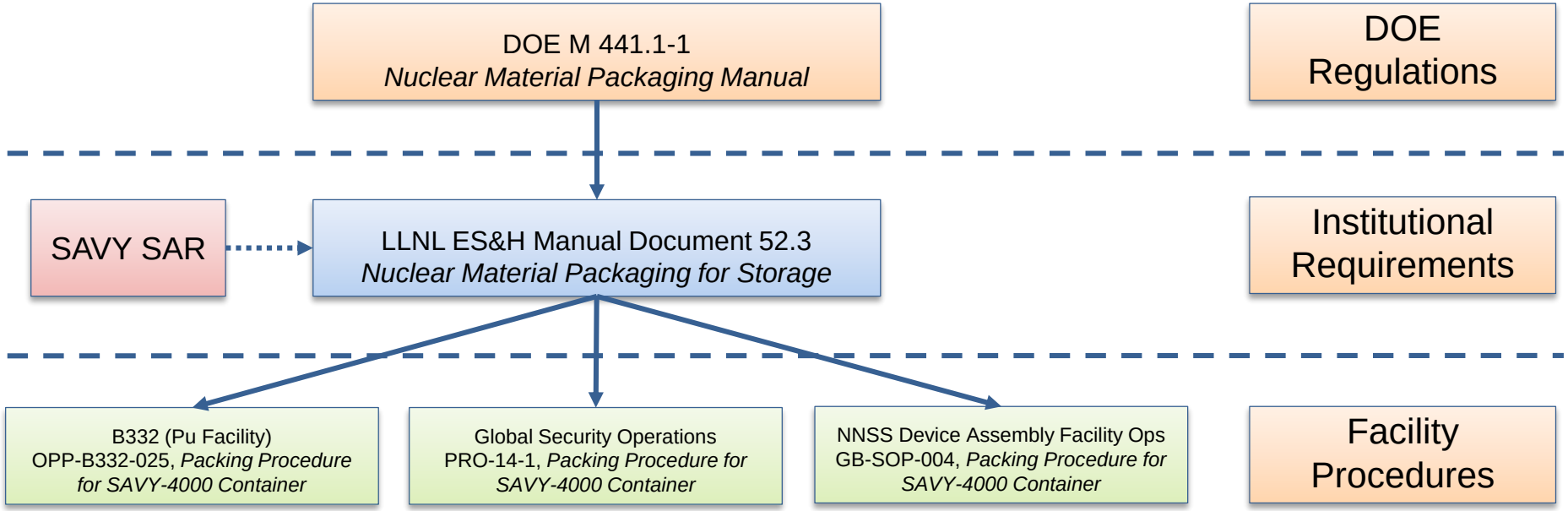
LLNL-PRES-787084
This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract DE-AC52-07NA27344. Lawrence Livermore National Security, LLC

Lawrence Livermore National Laboratory

Outline

- Implementation Flow Down of DOE M 441.1-1
- Operational Use of SAVY Container
- Future Updates to Nuclear Material Packaging Program

Implementation Flow Down of DOE M 441.1.-1



Operational Use of SAVY Container

Operational Use

- SAVY Utilization:
  - B332 (Pu Facility)
    - Vault Storage Containers (under DOE M 441.1-1)
    - Transport Containers (outside of DOE M 441.1-1)
  - Global Security Facilities
    - Storage of Sources Used in Detector Development/Testing (under DOE M 441.1-1)
  - Device Assembly Facility at NNSS
    - Storage of Material (under DOE M 441.1-1)
- SAVYs Undergo Formal Documented Inspection Prior to Release for Service
- SAVYs are utilized outside gloveboxes as tertiary containers
- All In-Scope Material at LLNL is Stored in SAVY Containers
- During Packaging/Unpackaging SAVYs Require Visual Inspection for Damage
- No Issues Have Been Reported with SAVY Container Use at LLNL

Future NMP Updates/Activities at LLNL

- Incorporation of Increased SAVY Design Life
  - Received 1-year extension on existing SAVY life from LFO
    - Extended from Mar 31, 2019 to Mar 31, 2020
  - ESH Document 52.3 update in progress to incorporate extended design life
    - Facility procedure updates will follow as necessary
- LANL SAVY DA Visit Sep 10-12
  - LANL will visit to discuss use of SAVY at LLNL as Secondary User

Y-12 National Security Complex

Managed and Operated by
Consolidated Nuclear Security, LLC

Nuclear Material Packaging Working Group Meeting
Y-12 Implementation Update for DOE M 441.1-1 Container

Craig Simon
Craig.Simon@cns.doe.gov
Design & SARP Support Lead

Y12 DOE M 441.1-1 Containers: Introduction

Background

- Funded FY 2012, initiated designs, and tested prototypes, late summer 2015
- NPO approved the Y-12 IP for DOE M 441.1-1, Rev. 5, 8/12/15
- Submitted Draft Safety Basis Document of the M441Storage Containers with Uranium-Bearing Content, SAR 802228-0001 000 01, to OPT for Review, August 2017
- Received Approved Safety Evaluation Report for the M441 Storage Containers with Uranium-Bearing Content, Rev. 0, March 2019

Y12 DOE M 441.1-1 Containers: Container types

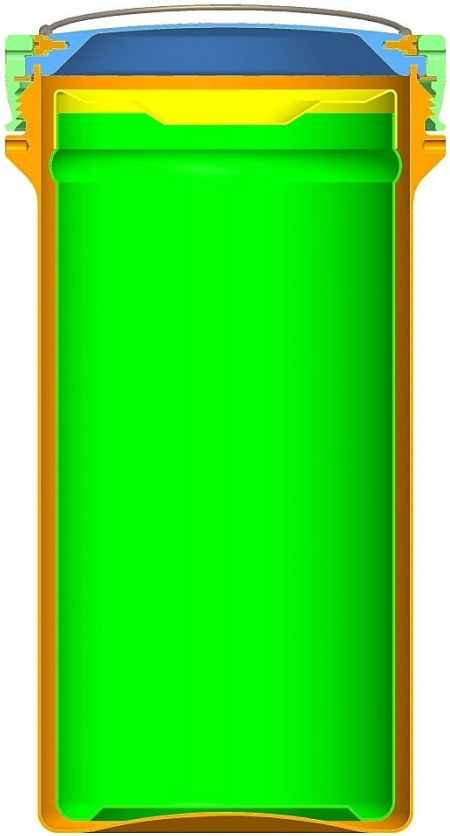
- Inner cans – ICC-03, -04, & -05, Light gauge stainless steel, easy to handle, easy to clean, and less costly
  - Easily opened and closed (i.e., convenience containers) with slip fit lids
  - Has direct contact with the nuclear material
  - Primarily manipulated within the confines of engineered containment barriers (e.g., gloveboxes)
  - Easily maneuvered for outer container loading
  - Not included in technical safety basis document
- Outer containers – M441-03, -04, & -05 Robust stainless steel design, tested and implemented according to the manual
  - Free of pinch points, burrs, sharp points, edges, and corners
  - Allows operator to ergonomically hand-tighten the lid or use a mechanical device
  - Includes lifting handles for grasping, lifting, and transporting
  - M441-03 outer container is identical to M441-05 outer container, except shorter and has vented lid and O-ring
  - Lids are not interchangeable between the M441-03 and M441-05 outer containers

Y12 DOE M 441.1-1 Containers: Inner Convenience Cans ICC-05 and ICC-03

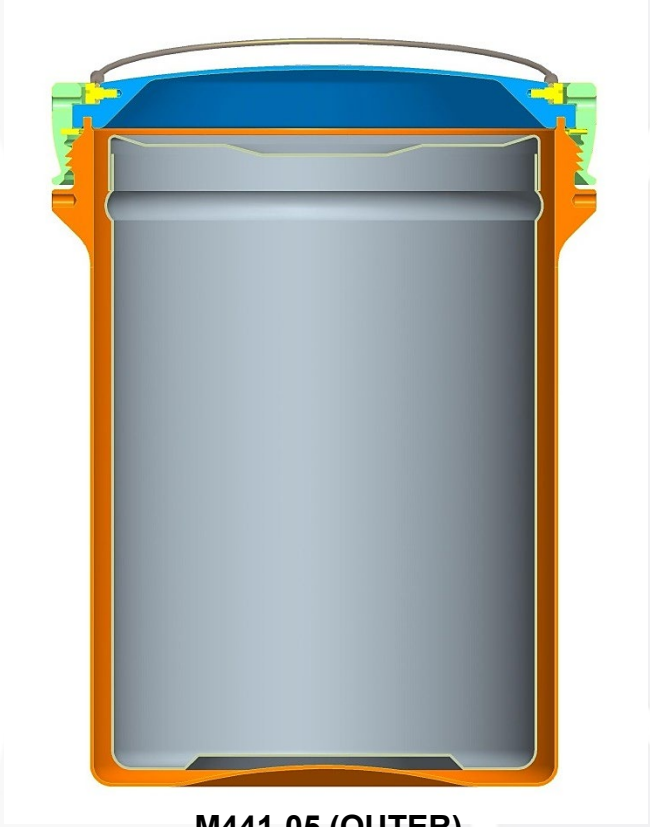
Y12 DOE M 441.1-1 Outer Containers: M441-04, M441-05, M441-03

Y12 DOE M 441.1-1 Containers: Outer with ICC Inner

M441-03 (OUTER)
WITH ICC-03
(INNER)

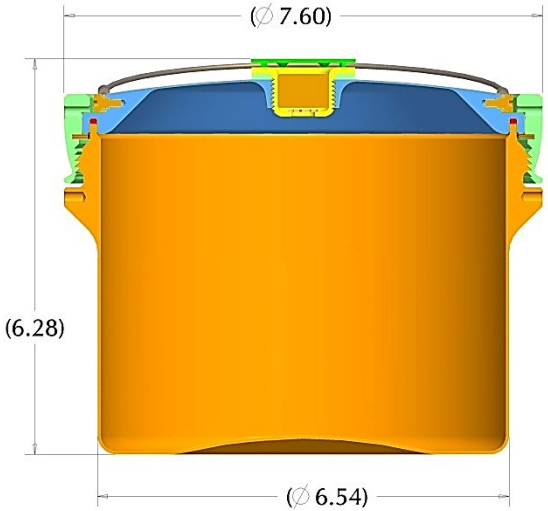


M441-04 (OUTER)
WITH ICC-04
(INNER)

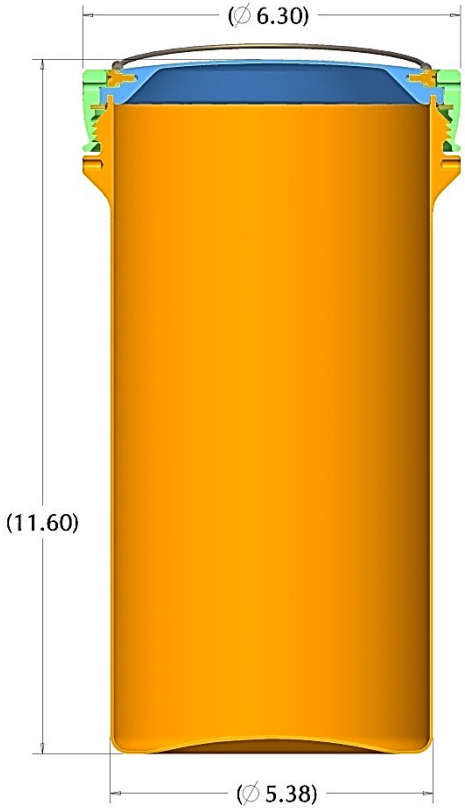


M441-05 (OUTER)
WITH ICC-05
(INNER)

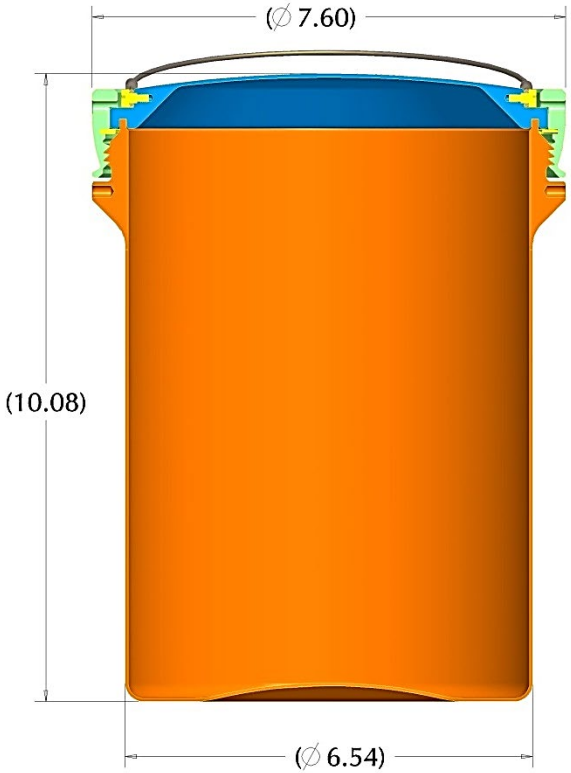
Y12 DOE M 441.1-1 Outer Container Dimensions



M441-03
WITH LID

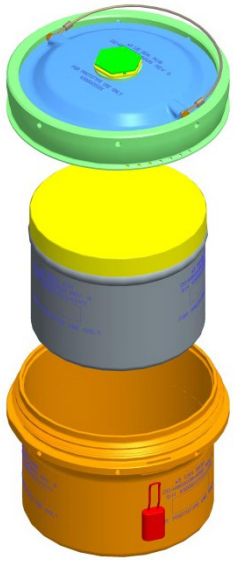


M441-04
WITH LID



M441-05
WITH LID

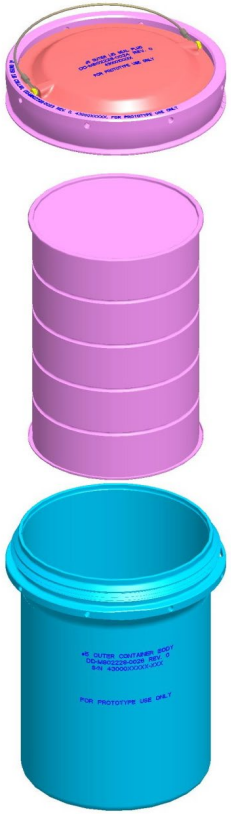
Y12 DOE M 441.1-1 Containers: Configurations



M441-03 OUTER CONTAINER WITH INNER CAN

M441-04 OUTER CONTAINER WITH INNER CAN

M44105 OUTER CONTAINER WITH INNER CAN



M441-05 OUTER CONTAINER WITH EXISTING CRIMP SEAL CAN

Y12 DOE M 441.1-1 Containers: Description (cont.)

- M441-03 outer container – ICC-03 inner can holding respirable oxides, chips, fines, slurries, sludges, solutions, and miscellaneous wet uranium-bearing materials; vented container in conjunction with inner can
- M441-04 outer container – small off-site customer-requested shipping container
- M441-05 outer container – ICC-05 inner can holding logs, briquettes, dry broken metal, gum drops, buttons, respirable oxides, powders, pin stock, slugs, and existing stainless steel crimp-sealed can used in existing Y-12 facilities
- ICC-03 inner can (with slip-fit lid) – chips, fines, slurries, sludges, solutions, and miscellaneous wet uranium-bearing materials
- ICC-04 inner can (with slip-fit lid) – logs, briquettes, dry broken metal, gum drops, buttons, respirable oxides, powders, pin stock, and slugs
- ICC-05 inner can (with slip-fit lid) –buttons, respirable oxides, powders, pin stock, and slugs

NOTE: The existing stainless steel crimp-sealed cans designed for storage in RCSBs will also fit into the M441-05 outer container for transport and handling.

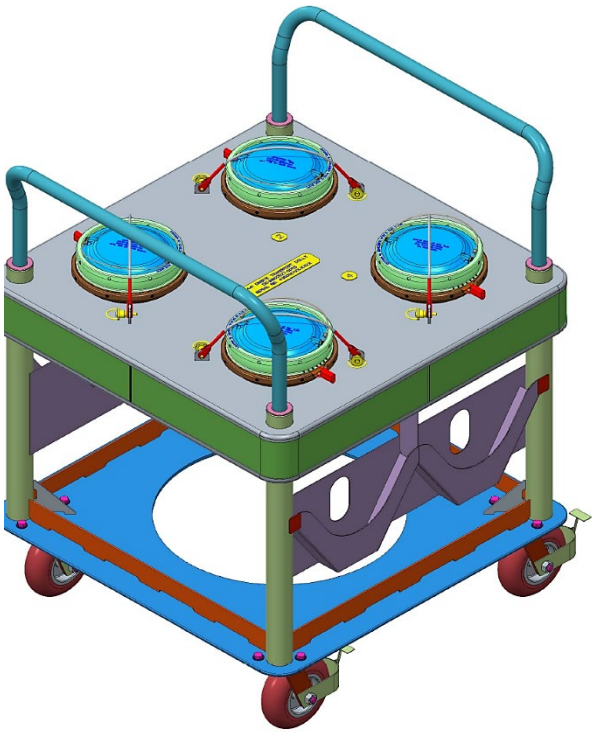
Y12 DOE 441.1-1 Containers: Description (cont.)

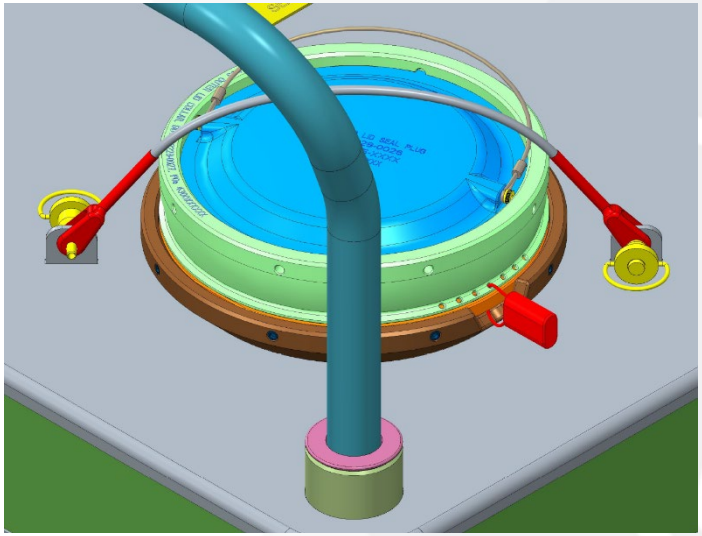
- Two dry material container designs (M441-04 and M441-05) will be used for 99+% of Y-12 storage.
  - Dry material containers are specifically designed for uranium materials for long-term storage
    - no degradable components over 50 years (all metal)
    - Sized to maximize uranium/can and UPF equipment
    - Designs are not leak-tight – will allow venting
  - One wet material container design (M441-03) will be used in applications where vent gases would require filtering.
    - not for long-term storage
    - contains degradable O-rings and filter elements
    - O-ring seal is liquid-tight

Y12 DOE 441.1-1 Containers: Container Advantages

- **Ease of use of the inner container inside of glove-boxes**
  - Slip-fit lid/Crimped Lid vs. Heavy Duty Screw Top Lid
  - Light weight can vs. Heavy Robust Storage Container (7.5 lbs.)
  - Life Cycle Cost- Inexpensive vs. Expensive
- **Interface and Transport with Existing Facilities and Containers**
- **Utilizes Existing Stainless Steel Crimp-lid Container (approved container)**
- **Close-fitting Outer Containers Meet the Nuclear Criticality Safety Requirements While Maximizing the Volume of the Inner Container**
- **Minimizes Y-12's Risk by Ultimately Reducing the Number of Containers Used in Production Areas**
- **Alleviates the Cost of Recontainerization for Long Term Storage**

Y12 DOE M 441.1-1 Containers: Onsite Transport Dolly

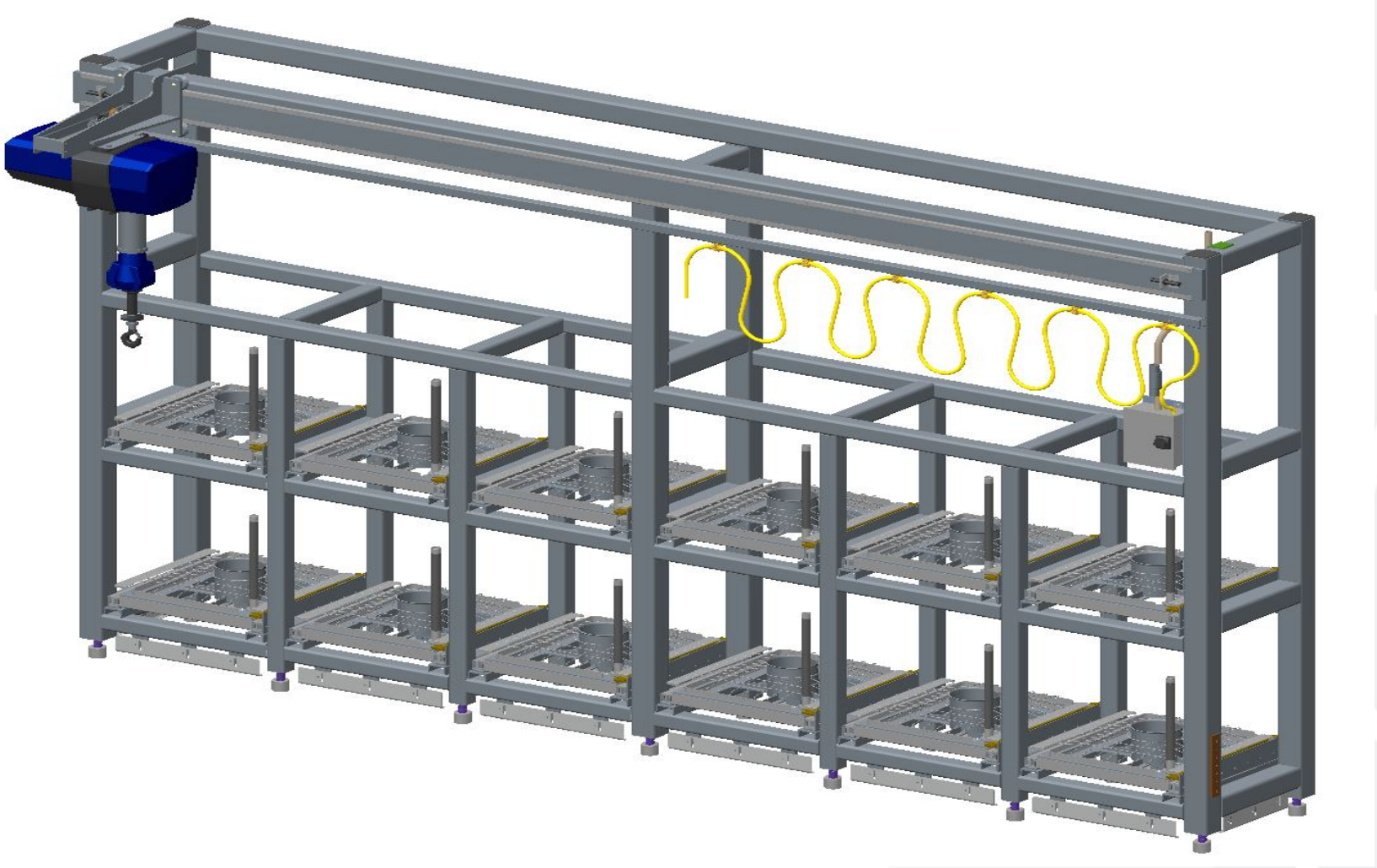




Note: The outers can be removed as shown above or affixed with set screws

Y12 DOE M 441.1-1 Containers: Onsite Transport Dolly

Y12 DOE 441.1-1 Containers: Storage Racks for Cans in UPF



Y12 DOE 441.1-1 Containers: Status

- **Safety Basis Document for the M441 Storage Containers with Uranium-Bearing Content, SAR 802228-0001 000 01: August 2017**
- **Receive Inner Convenience Cans ICC-03, ICC-04 May 2019**
- **Receive Outer M441-03, -04, & -05 Containers September 2019**
- **Implement into Facilities September/October 2020**

Questions - Contact Andrew Wysong – wysong1@llnl.gov – 925-422-3963

Lawrence Livermore National Laboratory

An NA-LA SME Perspective on DOE M 441.1-1 and draft STD

William Gordon,
NA-LA, Health Physicist
August 20th 2019

Bottom Line Up Front

- Regulatory ambiguity is not our ally in this situation.
  - I recommend that the standard include federal responsibilities before going into RevCom space.
- DOE Standards do not convey federal requirements, unless they are invoked in by CFR or a DOE Order.
  - DOE O 410.2, *Management of Nuclear Materials*, is the best candidate since it references (invokes) DOE M 441.1-1. I recommend that the standard be invoked by a DOE Order, if having a DOE O for nuclear material storage has already been ruled out.
- There are challenges when other M&O Partners and Field Offices rely on LANL's and NA-LA's processes, when the current manual requires each field office to approve the chemical characterization process.

Who I am

- DOE Radiation Protection Training Qualification Program Qualified SME.
- Provided guidance as part of NA-50 to NA-LA to approve LANL's surveillance program and reaffirmation of the prior approvals of the technical basis for the SAVY Series and Fuel Storage Outer Containers.
  - Surveillance program approved with NNSA-2018-002018
- Made technical recommendation to NA-LA to approve the life-extension request from 5 years to 15 years for the SAVY 4000 series containers (excluding 2 QT)
  - Approval Letter is in NNSA-2019-001918.

DOE M 441.1-1, *Nuclear Material Packaging Manual*

d. Field Element Managers.

(1) Review and approve the following contractor products:

(a) technical basis for nuclear material packaging systems including;

1 the evaluation of the chemical, radiological, and physical characteristics of the stored nuclear material

2 the nuclear material storage packaging designs

(b) nuclear materials packaging surveillance programs; and

(c) process for documenting its nuclear materials storage program.

(2) Ensure oversight of contractor's implementation of the requirements of this Manual.

(3) Ensure that a process exists for proposing, reviewing, approving, and implementing site corrective actions when necessary to ensure that the requirements of this Manual are met and to address conditions that are not protective of the public, workers, or the environment.

(4) Establish a risk-based prioritized schedule for implementing the nuclear material packaging requirements for material stored prior to the issuance of this Manual.

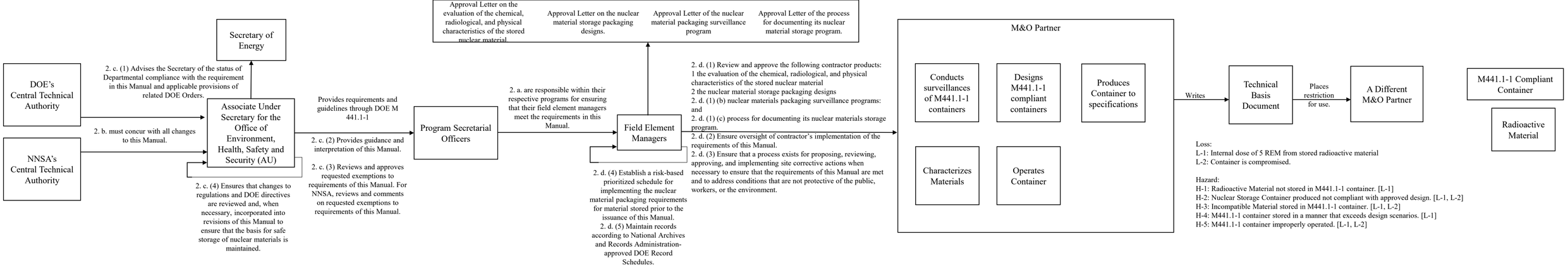
(5) Maintain records according to National Archives and Records Administration-approved DOE Record Schedules.

Issues NA-LA resolved/accepts

- Random Sampling – "Statistical sampling of packages for surveillance may be used for large, uniform populations of packages and/or contents." – DOE M 441.1-1

Unresolved Issues and Upcoming Opportunity

- **LANL needs a vehicle to submit updates to their technical basis document.**
  - Proposal, we treat the review of their technical basis document like an assessment, with a team composed of diverse SMEs.
  - Proposal accepted June 5th, 2019 by NA-LA
- **LANL has requested that the 2-Quart container be added to the SAVY 4000 series approved containers. SRS ARIES Project has a project milestone dependent on the 2-Quart container being approved.**
- **DOE M 441.1-1 is currently being revised into a technical standard and on track to be in RevCom in August.**
  - NA-LA is the only field office to have approve a nuclear material storage container compliant with the manual.


Secretary of Energy
DOE's Central Technical Authority
NNSA's Central Technical Authority
2. c. (1) Advises the Secretary of the status of Departmental compliance with the requirement in this Manual and applicable provisions of related DOE Orders.
2. b. must concur with all changes to this Manual.
Associate Under Secretary for the Office of Environment, Health, Safety and Security (AU)
2. c. (4) Ensures that changes to regulations and DOE directives are reviewed and, when necessary, incorporated into revisions of this Manual to ensure that the basis for safe storage of nuclear materials is maintained.
Provides requirements and guidelines through DOE M 441.1-1
2. c. (2) Provides guidance and interpretation of this Manual.
2. c. (3) Reviews and approves requested exemptions to requirements of this Manual. For NNSA, reviews and comments on requested exemptions to requirements of this Manual.
Program Secretarial Officers
2. a. are responsible within their respective programs for ensuring that their field element managers meet the requirements in this Manual.
Field Element Managers
2. d. (4) Establish a risk-based prioritized schedule for implementing the nuclear material packaging requirements for material stored prior to the issuance of this Manual.
2. d. (5) Maintain records according to National Archives and Records Administration-approved DOE Record Schedules.
Approval Letter on the evaluation of the chemical, radiological, and physical characteristics of the stored nuclear material.
Approval Letter on the nuclear material storage packaging designs.
Approval Letter of the nuclear material packaging surveillance program
Approval Letter of the process for documenting its nuclear material storage program.
2. d. (1) Review and approve the following contractor products:
1 the evaluation of the chemical, radiological, and physical characteristics of the stored nuclear material
2 the nuclear material storage packaging designs
2. d. (1) (b) nuclear materials packaging surveillance programs: and
2. d. (1) (c) process for documenting its nuclear materials storage program.
2. d. (2) Ensure oversight of contractor's implementation of the requirements of this Manual.
2. d. (3) Ensure that a process exists for proposing, reviewing, approving, and implementing site corrective actions when necessary to ensure that the requirements of this Manual are met and to address conditions that are not protective of the public, workers, or the environment.
M&O Partner
Conducts surveillances of M441.1-1 containers
Designs M441.1-1 compliant containers
Produces Container to specifications
Characterizes Materials
Operates Container
Writes
Technical Basis Document
Places restriction for use.
A Different M&O Partner
M441.1-1 Compliant Container
Radioactive Material
Loss:
L-1: Internal dose of 5 REM from stored radioactive material
L-2: Container is compromised.
Hazard:
H-1: Radioactive Material not stored in M441.1-1 container. [L-1]
H-2: Nuclear Storage Container produced not compliant with approved design. [L-1, L-2]
H-3: Incompatible Material stored in M441.1-1 container. [L-1, L-2]
H-4: M441.1-1 container stored in a manner that exceeds design scenarios. [L-1]
H-5: M441.1-1 container improperly operated. [L-1, L-2]