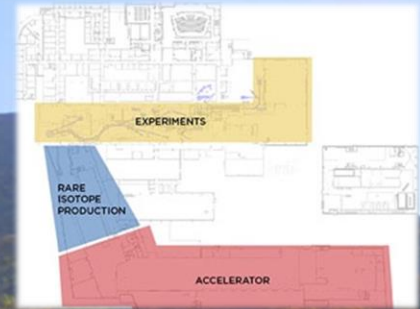




## *Long-Term Radiation Rugged Rotary Vacuum and Water Seals in Heavy-Ion Accelerators*

NanoSonic, Inc.: Dr. Jennifer Lalli  
Dr. William Harrison, Emma Sparks, Ben Woods, and Courtney Brand



DOE: Office of Nuclear Physics, Office of Science, U.S. Department of Energy

August 24, 2022 - DOE SBIR Phase IIA NP SBIR Exchange

TPOC: Dr. Michelle Shinn

# Overview

## Topic 26f: *Rotary Vacuum and Water Seals in Heavy-Ion Accelerators*

### Needed for NP Experiments:

- Ultra-high vacuum and water-cooled seals
- Constant rotation 600 rpm, 5,000 hr, ~1 year
- Extremely high annual radiation dose (~15 MGy)
- Need to change seal as infrequently as possible

### Partners:

- BNL – NSRL and BLIP
- MSU, FRIB
- Garlock
- Cardinal Rubber & Seal

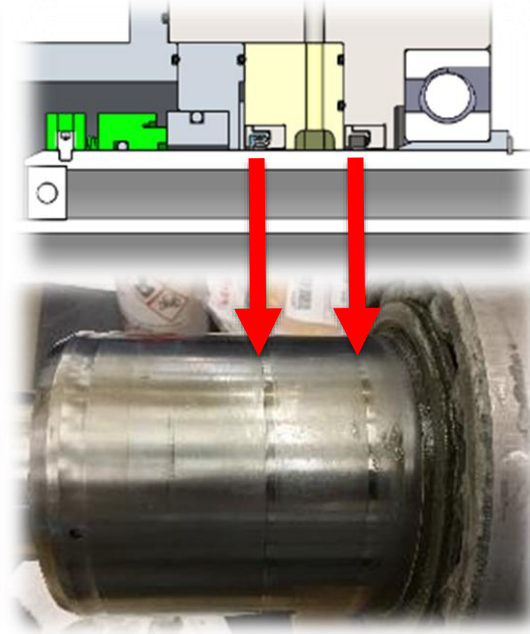
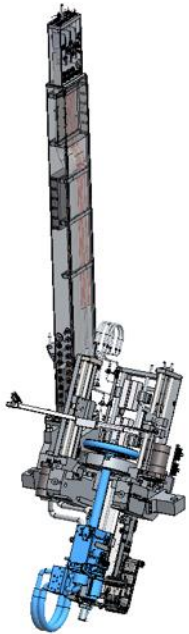


# DOE Topic 26f – Technology for High Radiation Environments

Grant # DE-SC0017107

## OBJECTIVE:

- Develop new rotary vacuum and water seals for rotating targets and beam dumps for rare isotope beam production and beam strippers in high-power heavy-ion accelerators
- Durable performance for 0.5 – 1.5 MGy/month, 1 year (5,000 hours), at 600 rpm over 32 °C to 66 °C, water side: 60 gpm (25 psi), vacuum side:  $1e^{-5}$  Torr L/s



*Need the mechanical performance of Teflon with enhanced Radiation & Less Abrasive  
Investigating new material for newly chosen design*



# NanoSonic Team

## & Our Commercial Partners/Investors



**Dr. Jennifer Lalli**, Chief Development Officer

Ph.D. Chemistry, Virginia Tech

- > 20 years of adhesive/sealant and gasket/seal development
- Implemented ExoStar Distribution of Products to Defense Primes
- 2 R&D 100 Awards for HybridSil® & Metal Rubber™ (issued patent)
- Commercialized 15 SBIR products sold at [www.nanosonic.com](http://www.nanosonic.com)



**Dr. William Harrison**, Gasket Production Lead

Ph.D. Chemistry, Virginia Tech

- >20 years of laboratory safety and production expertise
- Leads NanoSonic scale-up and product certification
- Commercializing Zero Humidity Fuel Cell Membranes with LANL



**Dr. Jie Wei**, Accelerator Systems Division Director - Michigan State University, **Facility for Rare Isotope Beams**

- Design, fabrication, installation, commissioning, and operations of all aspects of FRIB accelerator systems
- 27 years of research, management, and teaching experience on particle accelerators, major science projects, and major user facilities. design, research and development, construction, and commissioning of the Relativistic Heavy Ion Collider (RHIC), the interaction-region design of the Large Hadron Collider (LHC), the design, research and development, and construction of the Spallation Neutrino Source (SNS) ring, and the leadership of the China Spallation Neutron Source (CSNS) project.

**Jeongseog Song**, Target and Beam Dump Systems Group Leader

**Takuji Kanemura**, Drs. Philip Morrison, Michael Larmann, and Nicholas Reha



Facility for Rare Isotope Beams  
at Michigan State University

# NanoSonic is now ISO 9001:2015 Certified by NSF-ISR



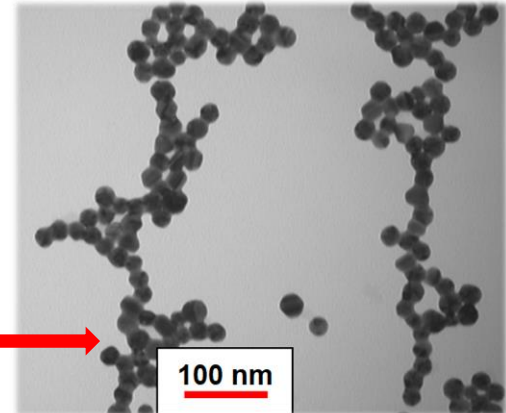
NSF-ISR

Registered to ISO 9001

## Development and Manufacturing of Novel Materials and Devices



250-gal, 55-gal, 1-10 L in hood, two 20L, and one 100 L reactor

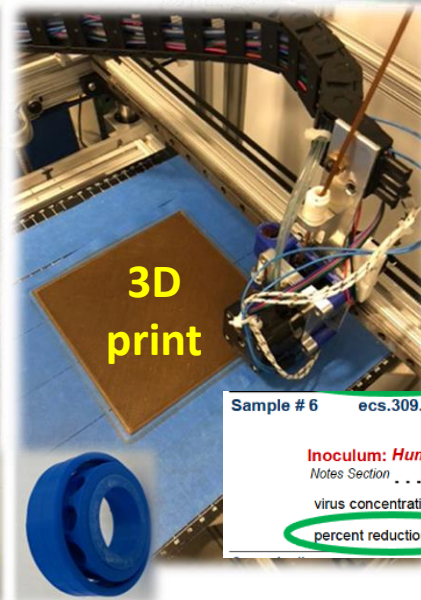
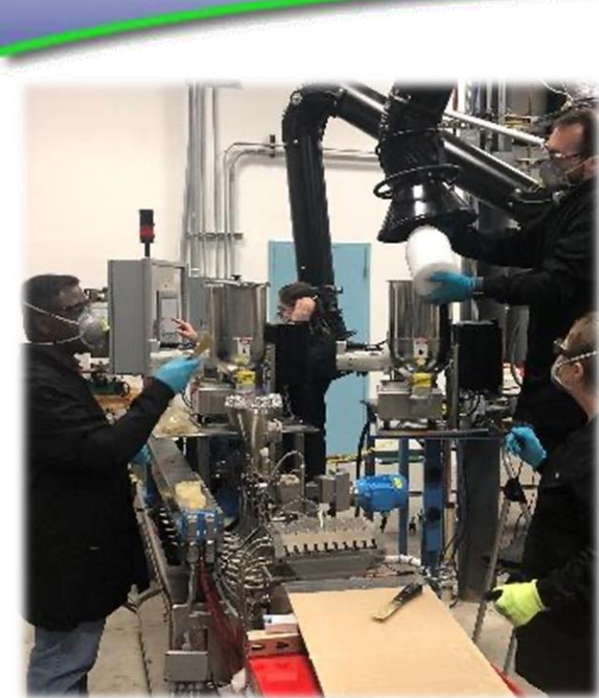


Au from 100 -L



# NanoSonic Production Capabilities:

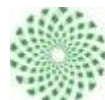
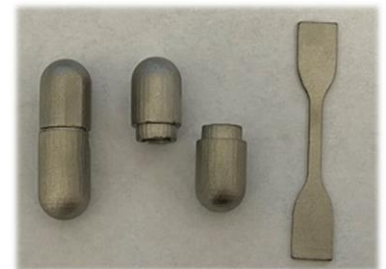
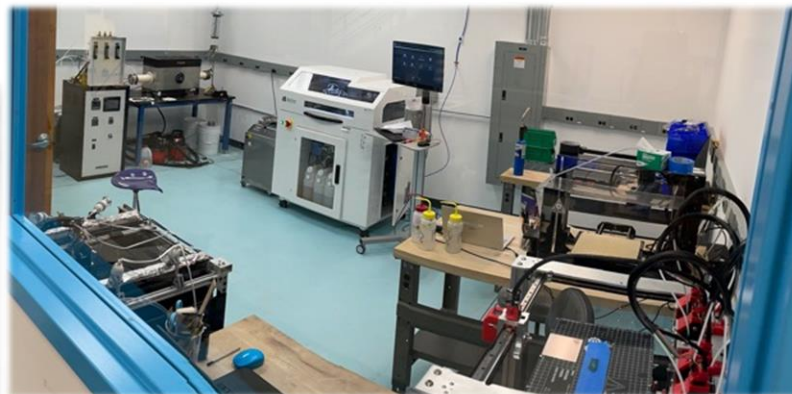
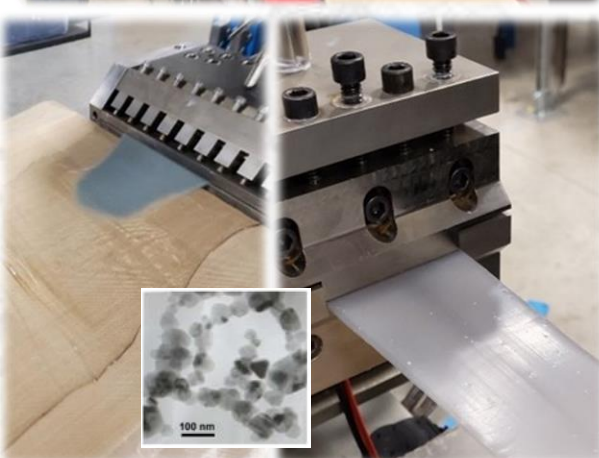
## Extrusion and 3D Printing of Radiation Tolerant Polymers, Metals, & Ceramics



Sample # 6 ecs.309.103.6

Inoculum: *Human coronavirus (OC43)*  
Notes Section .....

virus concentration  
percent reduction = 99.93



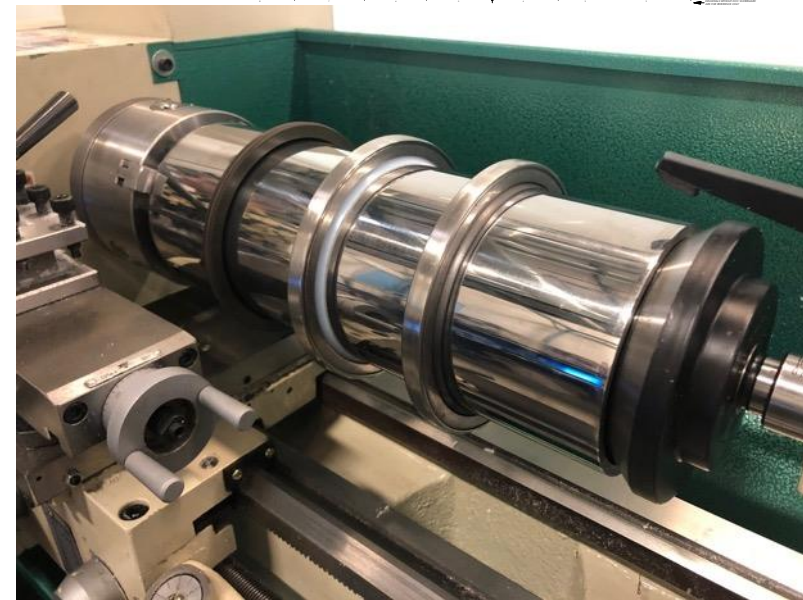
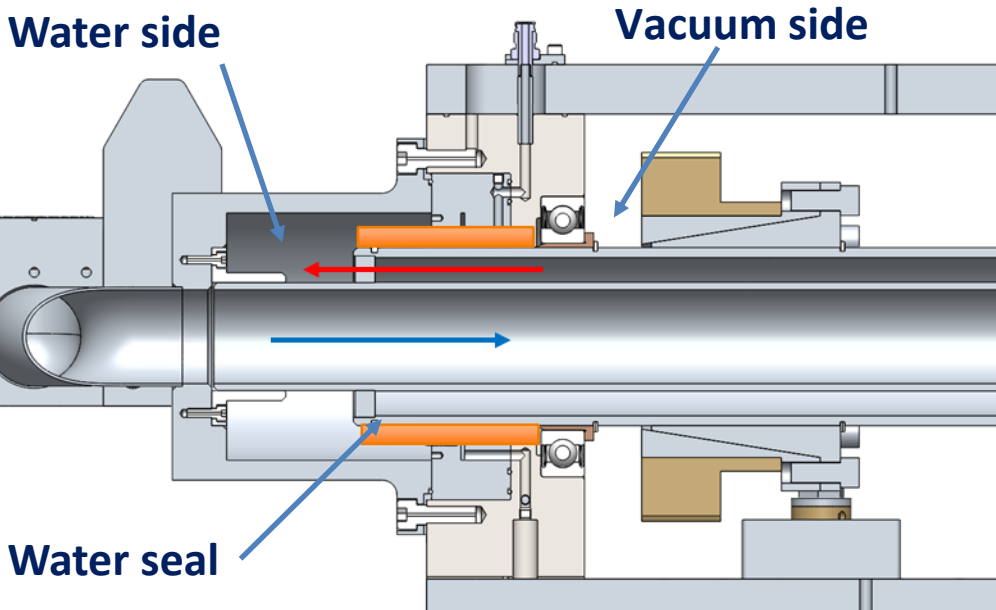
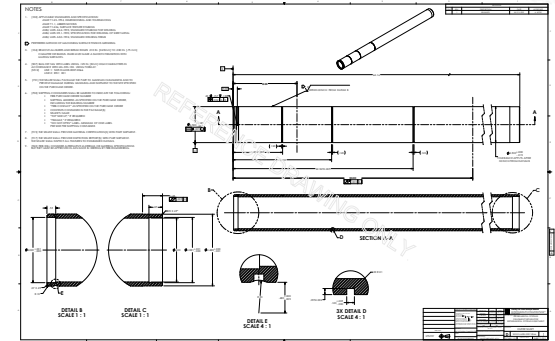
NanoSonic

# Goal:

## *Develop New Materials and Seal Designs for FRIB Beam Dump*

### GOALS:

- Develop new PTFE like polymers with radiation resistance
- Extrude compounded films not commercially available
- Implement new seal design – Flood-Gard® bearing isolator



*Reproduced 4.5" SS Shaft for Abrasion Testing of New Seal Materials to Mimic Beam Dump Water Seal*

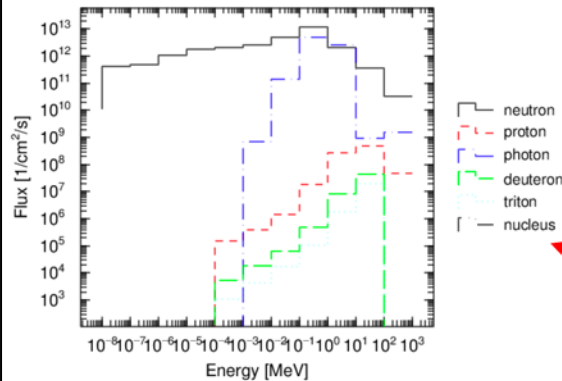


# High Dose Exposure

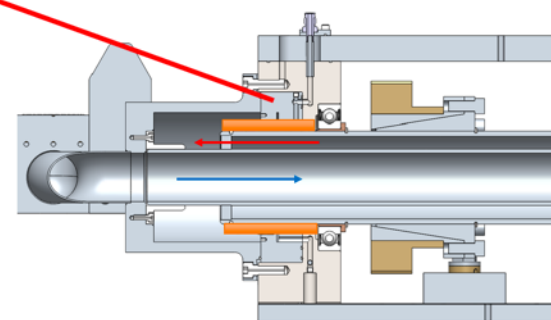
## Particle Energy Spectra for Beam Dump's Rotating Water Seal

Energy, MeV		Flux, particles/cm <sup>2</sup> /second				
E_low	E_high	Neutrons	Protons	Photons	Deuterons	Tritons
1.0E-09	1.0E-08	5.1E+06	0.0E+00	0.0E+00	0.0E+00	0.0E+00
1.0E-08	1.0E-07	1.9E+08	0.0E+00	0.0E+00	0.0E+00	0.0E+00
1.0E-07	1.0E-06	2.3E+08	0.0E+00	0.0E+00	0.0E+00	0.0E+00
1.0E-06	1.0E-05	4.9E+08	0.0E+00	0.0E+00	0.0E+00	0.0E+00
1.0E-05	1.0E-04	8.3E+08	0.0E+00	0.0E+00	0.0E+00	0.0E+00
1.0E-04	1.0E-03	9.7E+08	6.8E+01	0.0E+00	2.4E+00	5.0E-01
1.0E-03	1.0E-02	1.2E+09	1.8E+02	3.2E+05	8.3E+00	2.0E+00
1.0E-02	1.0E-01	2.3E+09	6.7E+02	6.7E+07	3.0E+01	7.9E+00
1.0E-01	1.0E+00	5.3E+09	8.2E+03	2.3E+09	2.3E+02	4.8E+01
1.0E+00	1.0E+01	9.5E+08	1.2E+05	1.2E+09	3.8E+03	8.1E+02
1.0E+01	1.0E+02	1.6E+08	2.3E+05	4.3E+05	2.0E+04	9.3E+03
1.0E+02	1.0E+03	1.5E+07	2.1E+04	7.3E+05	0.0E+00	0.0E+00

■ <sup>48</sup>Ca at 400 kW, 261 MeV/u → 40Mg



D. Georgobiani

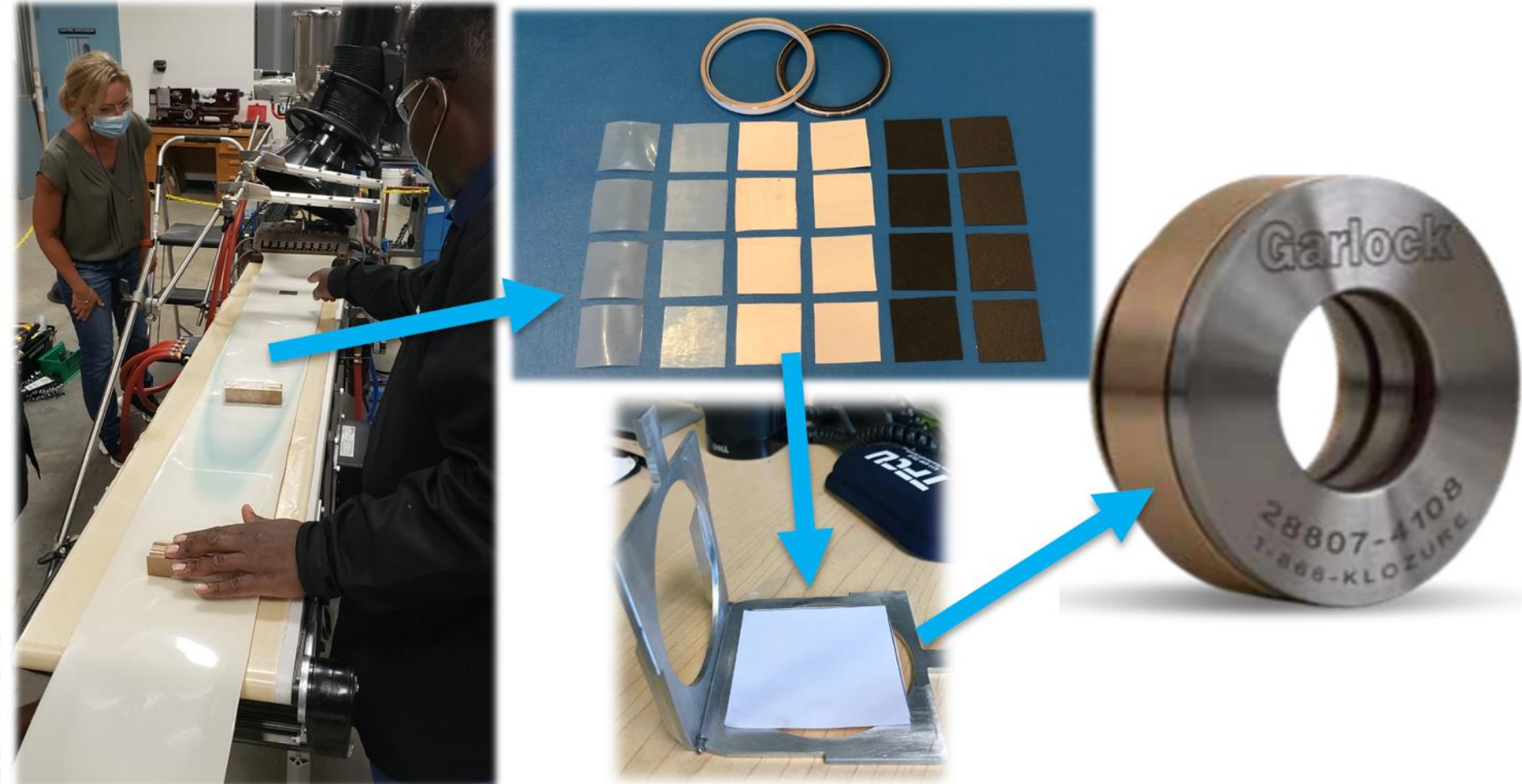


**High Energy 260 MeV when operated at 400 kW**



# Technical Approach

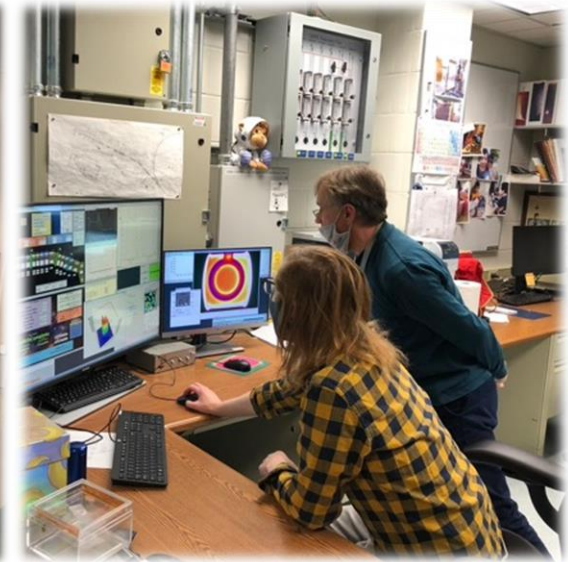
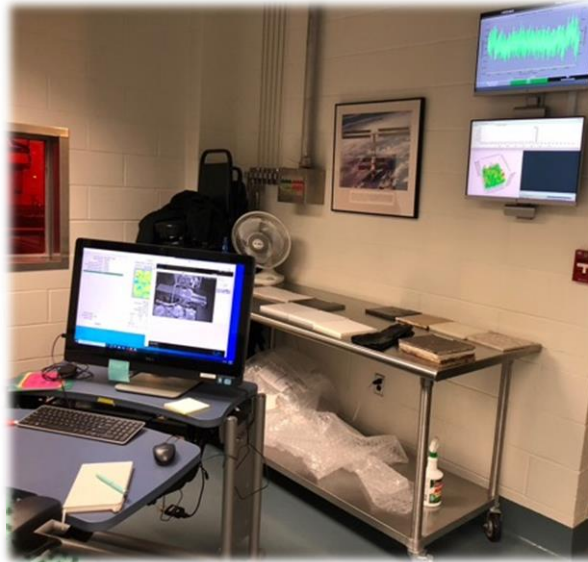
*Extrude New Compounded Materials for use in New Seal Designs*  
*Expose Materials to High Energy and High Dose Radiation for Durability Study*



*Extrude NanoSonic Modified Polymer for High Dose Exposure at BNL BLIP  
and Integration with Garlock Housing*

# Radiation Exposure Run 1 - May 7, 2021

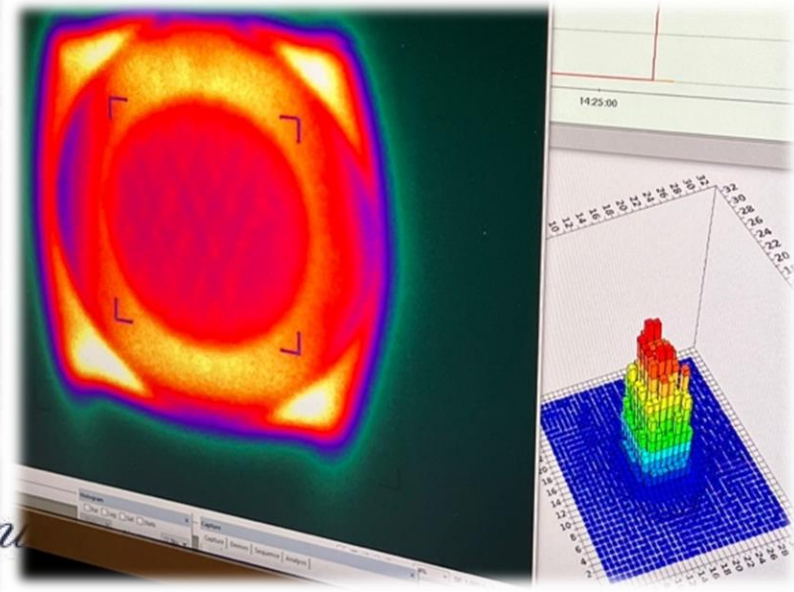
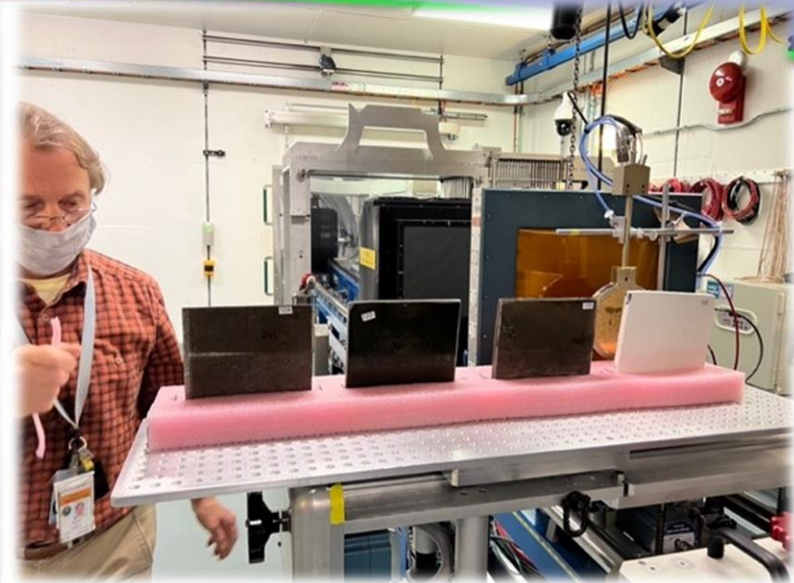
## High Energy – 1 GeV Fe / 1 GeV proton



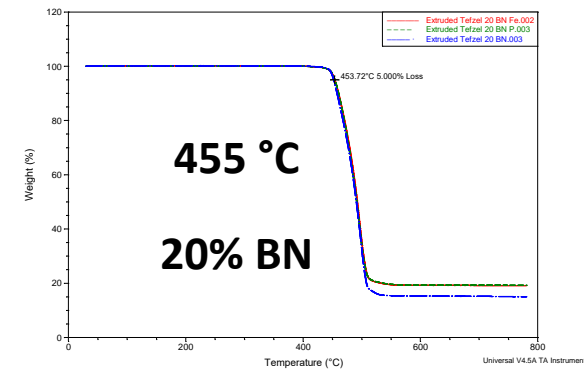
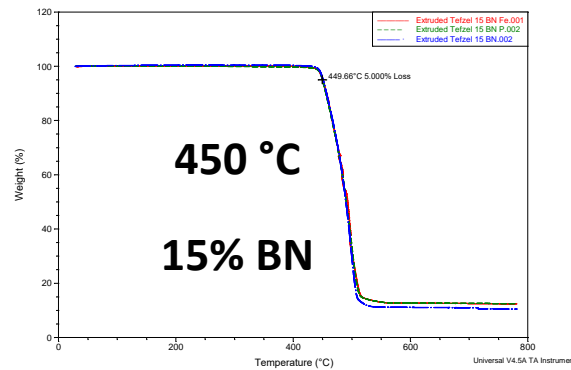
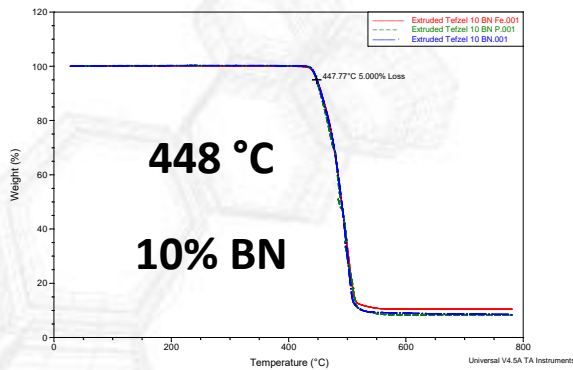
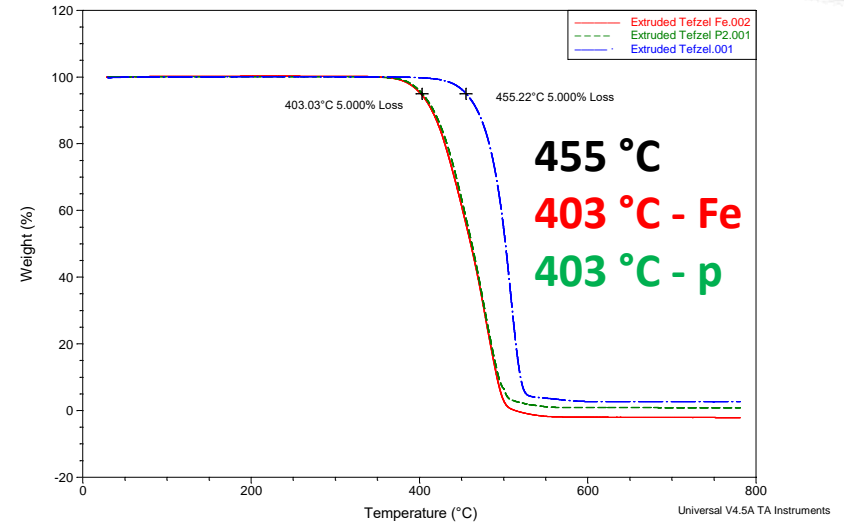
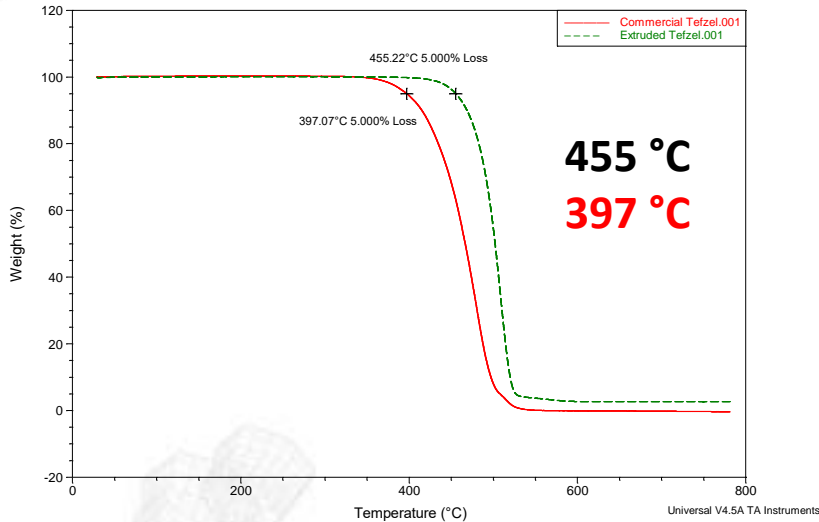


# Radiation Exposure Run 2 - June 13, 2022

## Lower Energy – 400 MeV Fe / 100 MeV proton



# TGA for COTS and NanoSonic Polymer pre- and post- 1 GeV Fe and 1 GeV proton

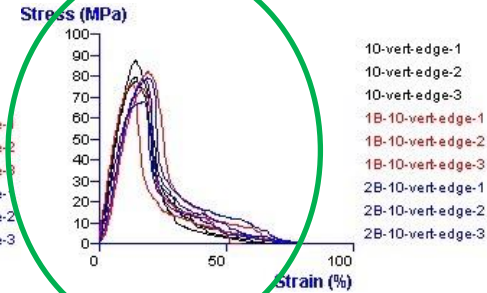
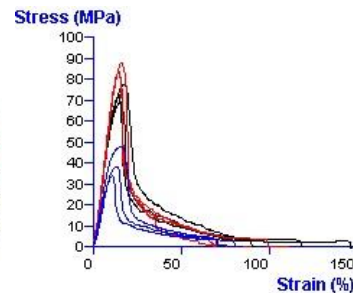
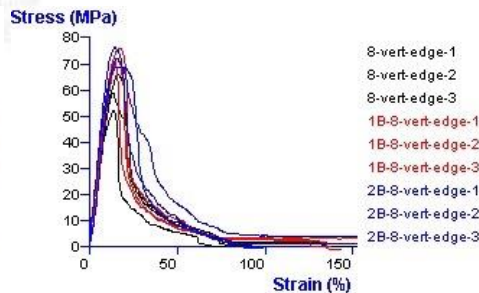
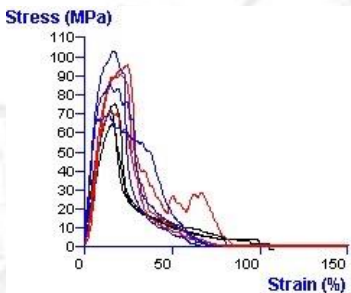
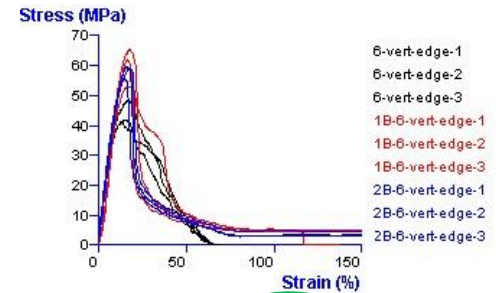
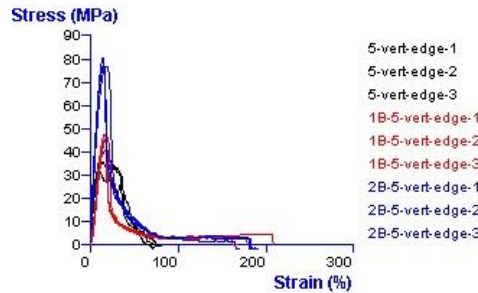
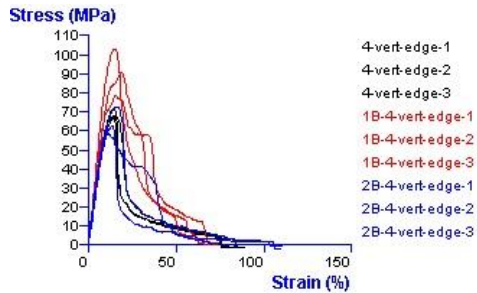
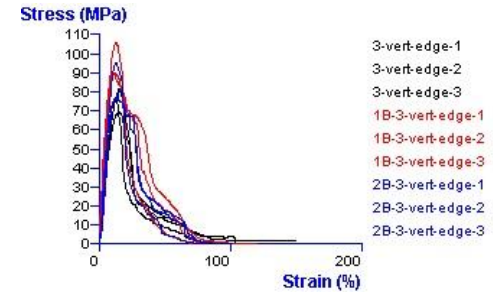
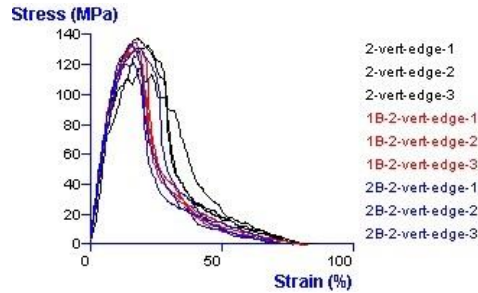
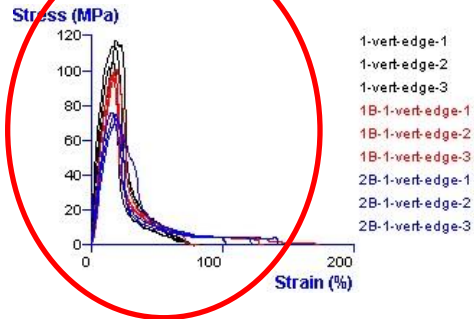


**NanoSonic-polymer ~60 °C increase over Commercial**

**Increasing BN Provides Increasing Enhancements in Radiation Resistance 45 - 52 °C increase**



# Pre- and Post- Irradiation – 27 Gy 1 GeV Fe and 1 GeV proton - separate

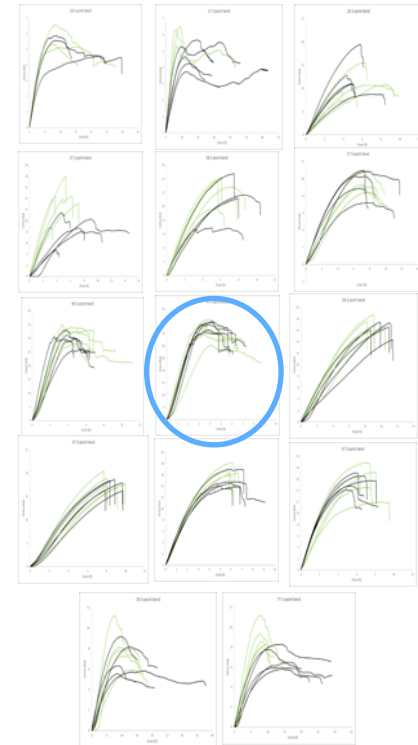
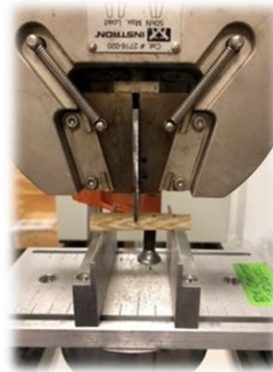
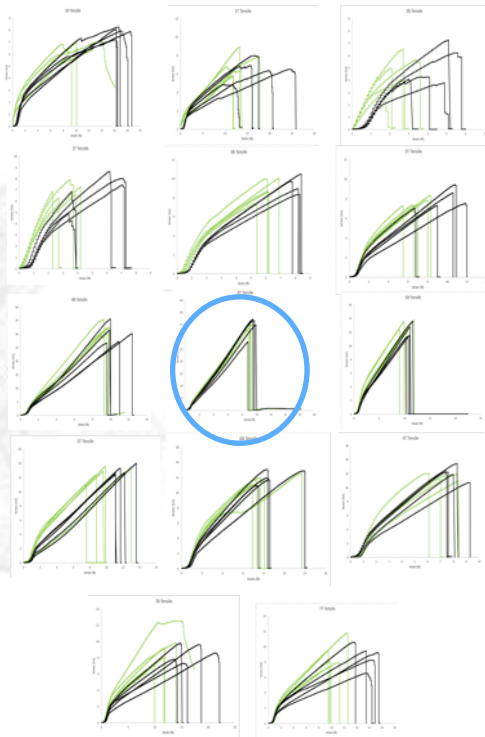


Note Tensile Strength Variations or Stability Post **Fe** and **Proton** Exposure

# Pre- and Post- Irradiation Tensile and 3-pt Bend

## 54 Gy dose: *1 GeV proton + 1 GeV Fe*

The composite sample was first irradiated using a beam of Fe ions at an energy of 1000 MeV/nucleon at the NASA Space Radiation Laboratory (NSRL) at Brookhaven National Laboratory. The beam was prepared with a uniform radiation field that spanned 20 x 20 cm<sup>2</sup>. The dosimetry was performed with a 1 cm<sup>3</sup> ion chamber with a NIST-traceable calibration for dose delivered in water. After calibration, the composite sample was placed on the beamline and exposed for 67.62 minutes for a total dose of 27 Gray. The beam came in “spills” that were ~400 milliseconds long with a period of 6.6 seconds. The sample was then irradiated with a proton beam of 1000 MeV energy where the exposure of 27 Gray took 24.36 minutes.

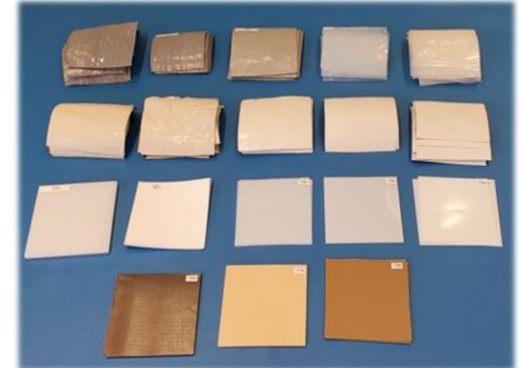
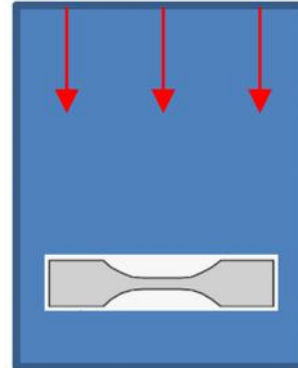
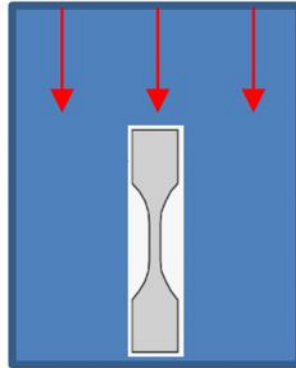


***Composites with High-Z Layer Exhibits Trend for Enhanced Durability***

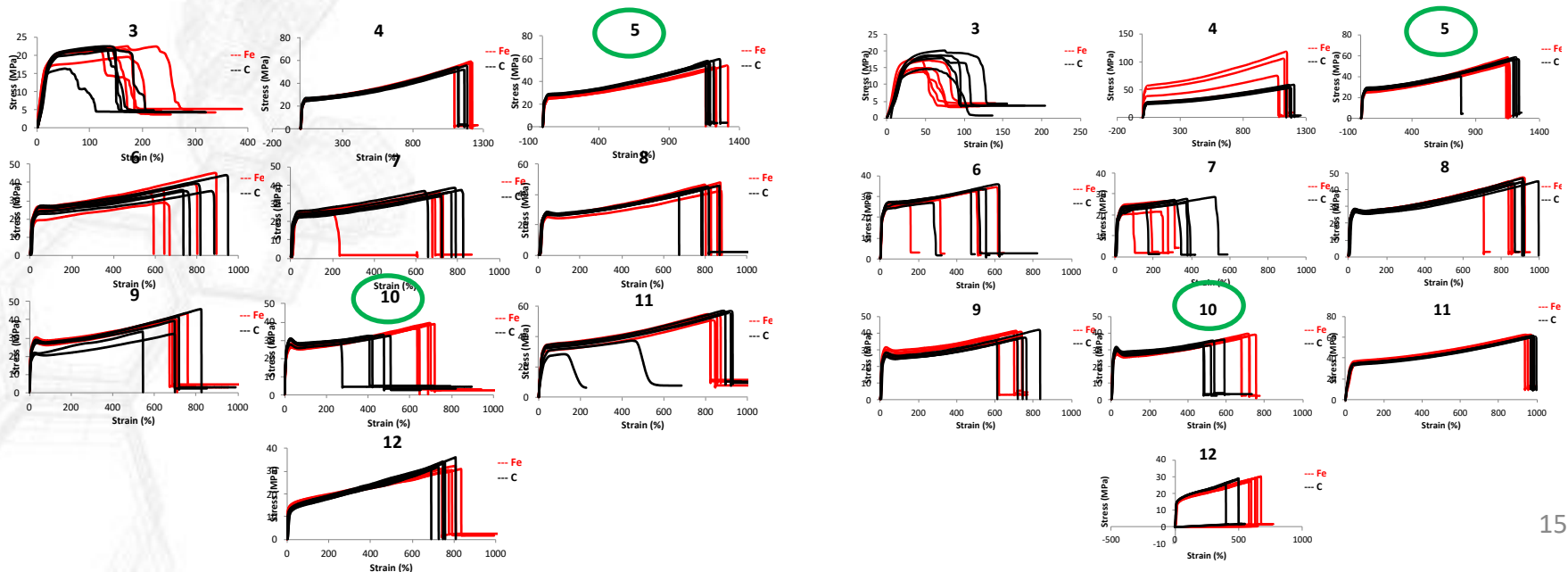


# Increased Dose – 214 Gy

## Exposure at NSRL to Fe 1 GeV for Down-Selection



### Mechanical Properties for All NanoSonic Films in Parallel and Perpendicular Directions



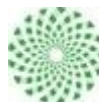
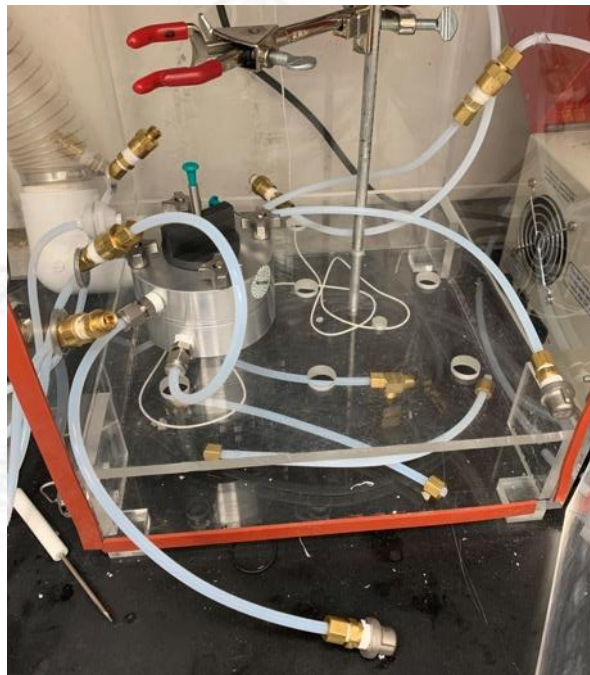
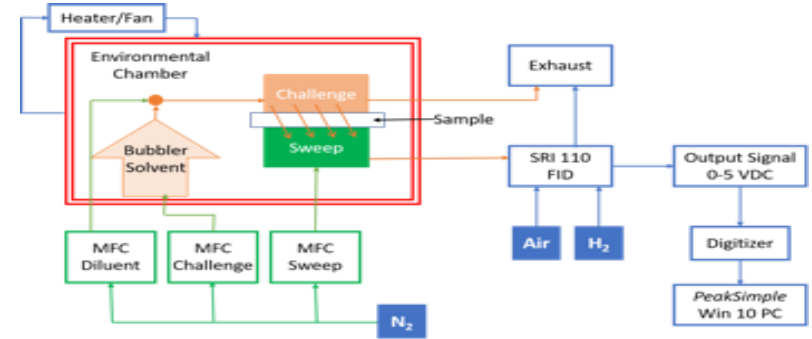
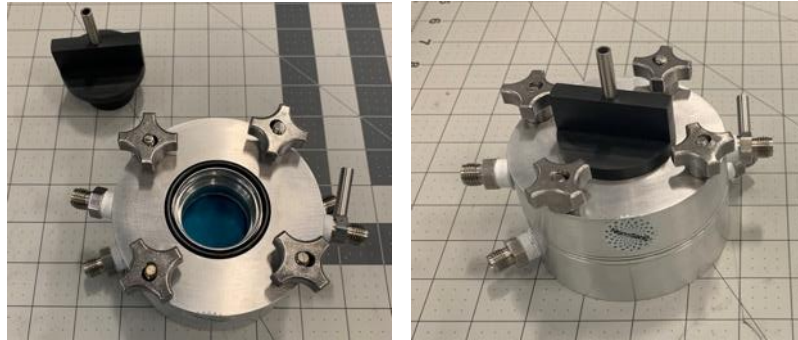
# Permeation Testing at NanoSonic Before and After Radiation

I / II

Chemical permeation resistance: NFPA 1994 Class 1

20 g/m<sup>2</sup> challenge over 1 hour

20 g/m<sup>2</sup> challenge over 6 hours





# Permeation Testing at Intertek for Related Materials – CBRN Gloves

Intertek and Avarint results verify that Dimethyl sulfate, Soman (GD), and Distilled Mustard (HD) are “non detectable” when tested for 60 min against NanoSonic’s FKM at a thickness of 0.23mm

## SECTION 7.1.2.1

### LIQUID TOXIC & GASEOUS INDUSTRIAL CHEMICAL PERMEATION RESISTANCE TEST

CONDITIONING: In Accordance with Section 8.7.7.1.1

<b>CHALLENGE CHEMICAL:</b>	Dimethyl sulfate	<b>CONCENTRATION:</b>	20 g/m <sup>2</sup>
<b>SAMPLING METHOD:</b>	Universal Sorbent Tube	<b>ANALYSIS METHOD:</b>	GC-MS
<b>MINIMUM DETECTION LIMIT:</b>	0.20		

TEST RESULTS	SAMPLE 1	SAMPLE 2	SAMPLE 3	AVERAGE
Cumulative Permeation Mass (µg/cm <sup>2</sup> ) - T 0-15	<0.20	<0.20	<0.20	<0.20
Cumulative Permeation Mass (µg/cm <sup>2</sup> ) - T 15-60	<0.20	<0.20	<0.20	<0.20
Cumulative Permeation Mass (µg/cm <sup>2</sup> ) - T 0-60	<0.20	<0.20	<0.20	<0.20



**Avarint** NIPA TEST REPORT

Method: NPPA 1994, Class 1 (2018 ed) Customer: Intertek, 3933 US Route 1, Cortland, NY 13045  
 Material ID: HANONER CRT21092047-001 G204792074

Material Type: Tan M4335-131 Fabric (P)

Test Date: 6/22/2021 Report Date: 6/30/2021

Test Trial Summary Information: Test Trial: A0226-003, Chemical: **DM**, DCR Response: 101%, Relative Humidity: 71.8%, Detection Limit: 0.1 µg/cm<sup>2</sup>

Test Results:

Avarint Sample Control Number	Sample Type	Permeation (µg/cm <sup>2</sup> )	Avarint Sample Control Number	Sample Type	Permeation (µg/cm <sup>2</sup> )
		t = 15 min t = 60 min			t = 15 min t = 60 min
A0226-0012	Replicate 1	ND ND			
A0226-0016	Replicate 2	ND ND			
A0226-0017	Replicate 3	ND ND			
A0226-0018	Slip Cal	ND ND			
Pos. Cal	Average recovery (%)	97%			

**Avarint** NIPA TEST REPORT

Method: NPPA 1994, Class 1 (2018 ed) Customer: Intertek, 3933 US Route 1, Cortland, NY 13045  
 Material ID: HANONER CRT21092047-001 G104792074

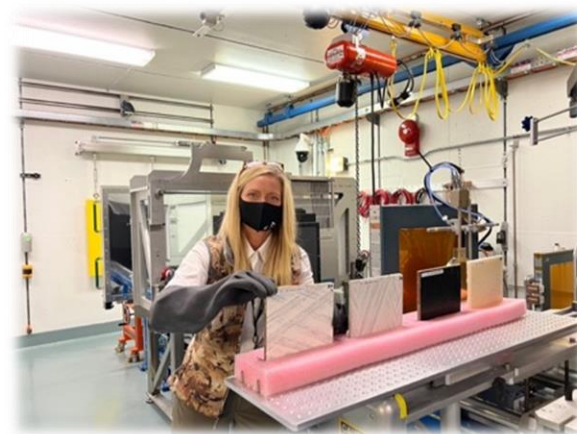
Material Type: Tan M4335-131 Fabric (P)

Test Date: 6/22/2021 Report Date: 6/30/2021

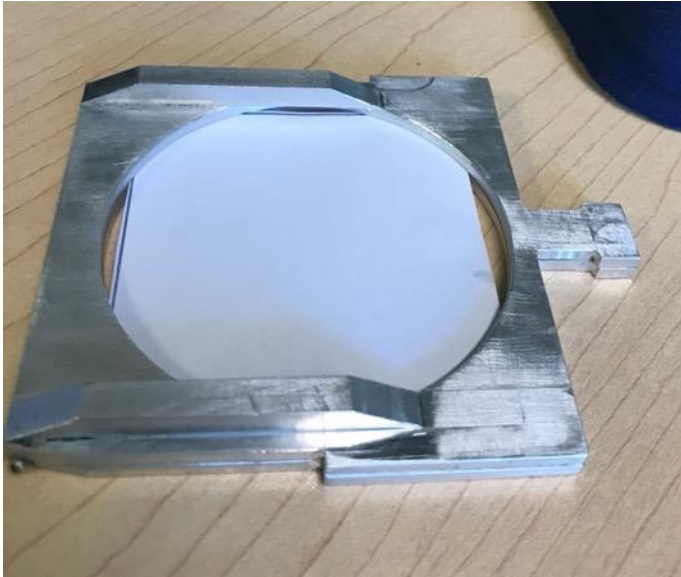
Test Trial Summary Information: Test Trial: A0226-004, Chemical: **GD**, DCR Response: 100%, Relative Humidity: 79.0%, Detection Limit: 0.05 µg/cm<sup>2</sup>

Test Results:

Avarint Sample Control Number	Sample Type	Permeation (µg/cm <sup>2</sup> )	Avarint Sample Control Number	Sample Type	Permeation (µg/cm <sup>2</sup> )
		t = 15 min t = 60 min			t = 15 min t = 60 min
A0226-0019	Replicate 1	ND ND			
A0226-0020	Replicate 2	ND ND			
A0226-0021	Replicate 3	ND ND			
A0226-0022	Slip Cal	ND ND			
Pos. Cal	Average recovery (%)	96%			



# High Dose Exposure at BNL BLIP



Dr. Dmitri Medvedev  
Dr. Cathy Cutler  
Dr. Dohyun Kim

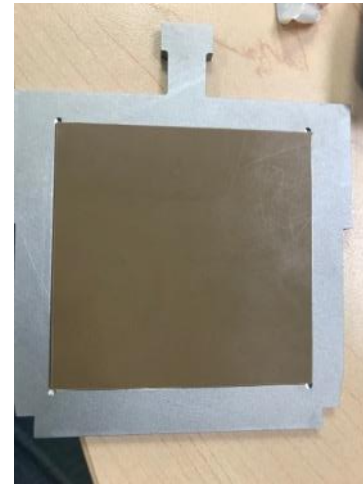
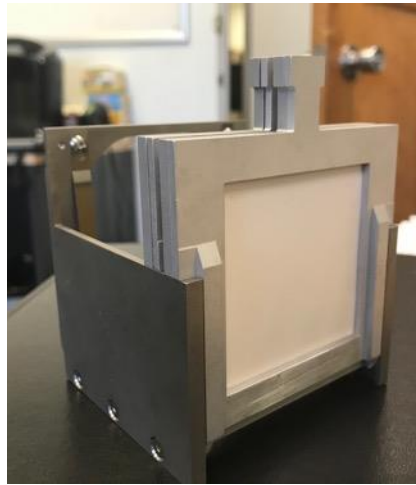
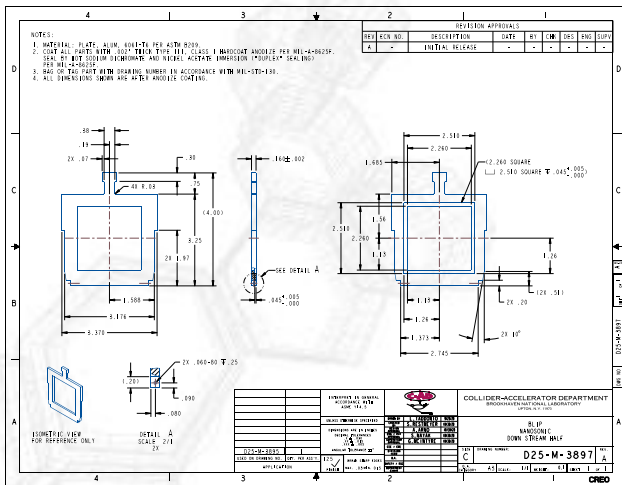
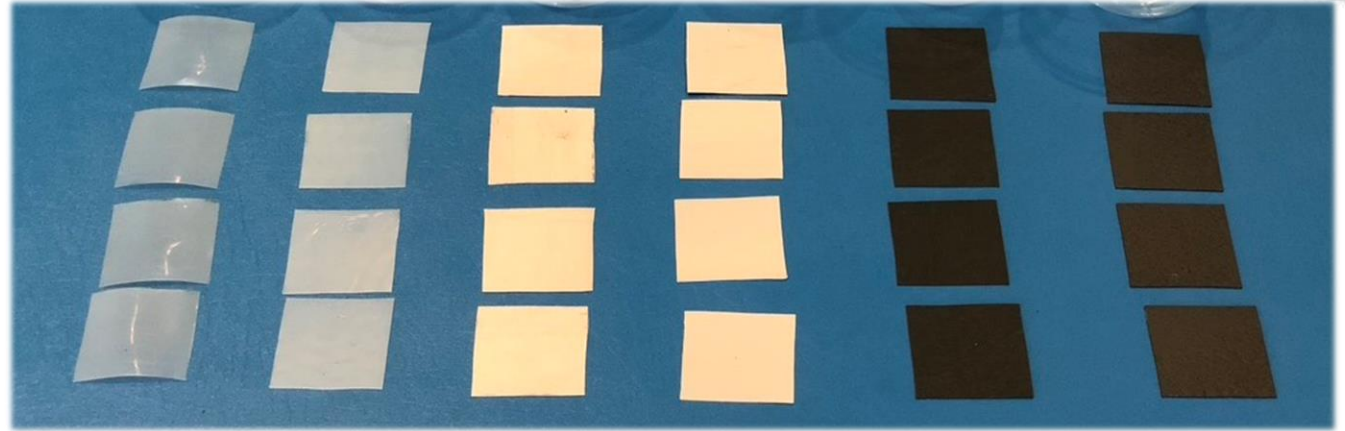
Three rotary feedthroughs were immersed in cold water and irradiated under static conditions at the Brookhaven Linac Isotope Producer (BLIP) at the Brookhaven National Laboratory (BNL). An 112 MeV proton beam was stopped in several thick solid isotope production targets made of rubidium chloride and gallium and provided the feedthrough radiation absorbed dose. The average absorbed dose rate was 2.24 MGy/day for different radiation doses: 0.2, 2 and 20 MGy. The radiation consisted primarily of fast neutrons (mean energy 8.5 MeV), protons (mean energy 20 MeV), gamma rays and electrons. These radiation types, their intensity and energy are close to the ones expected under FRIB conditions in the target and beam dump systems area.

0.2 MGy ~ 2h  
2 MGy ~ 21 h  
20 MGy ~ 9 days

***Run Scheduled upon Targetry Housing Construction***



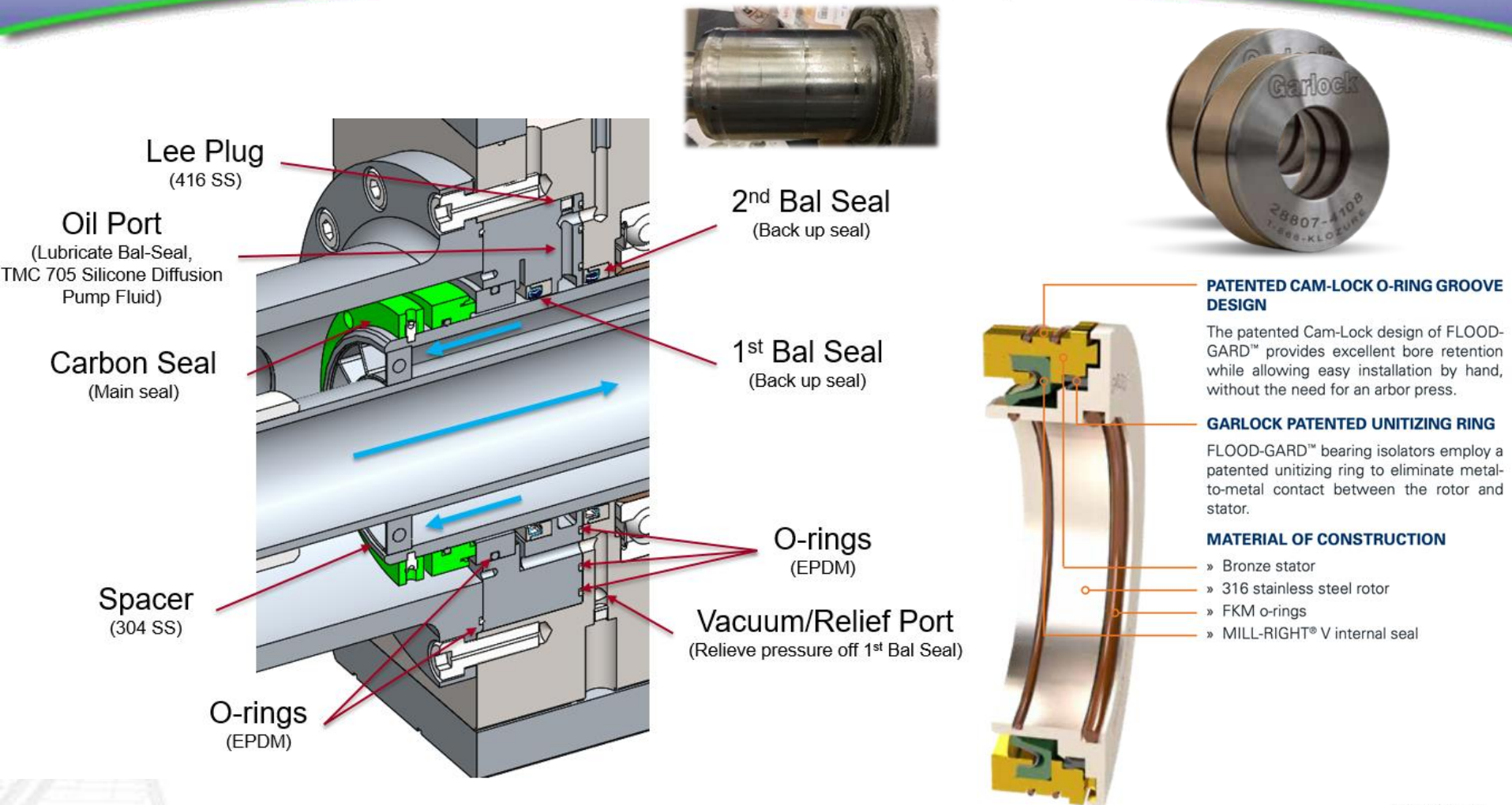
# High Dose Exposure at BNL BLIP Extruded and Delivered Films



Run Scheduled for 2022

# New Design and Materials:

## Develop New Materials for Pseud-Flood-Gard Design

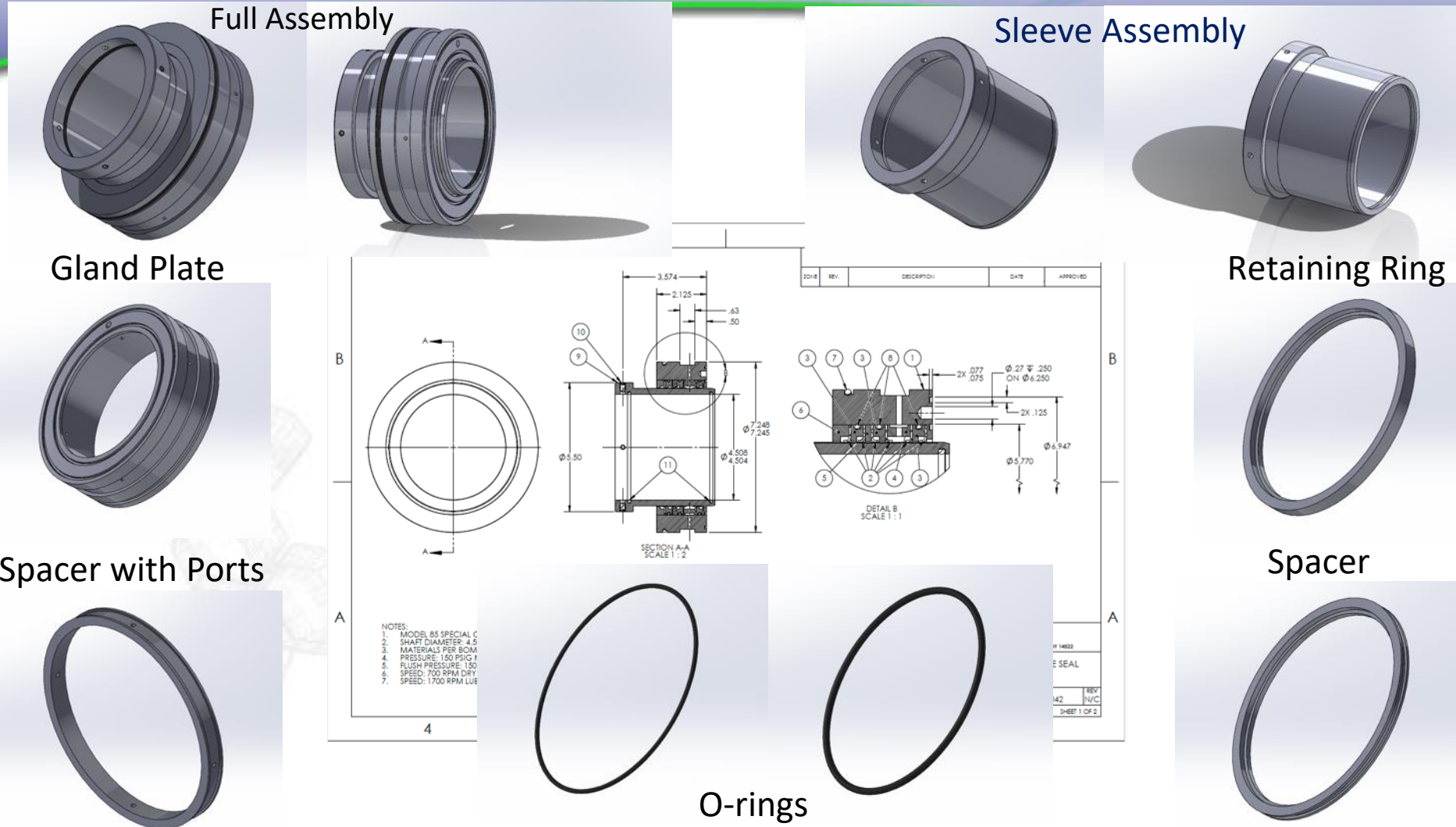


**Delivering Flood-Gard Commercial Seals and NanoSonic Modified Seals  
o-rings, unitizing ring, and V internal seal**



# Final Design

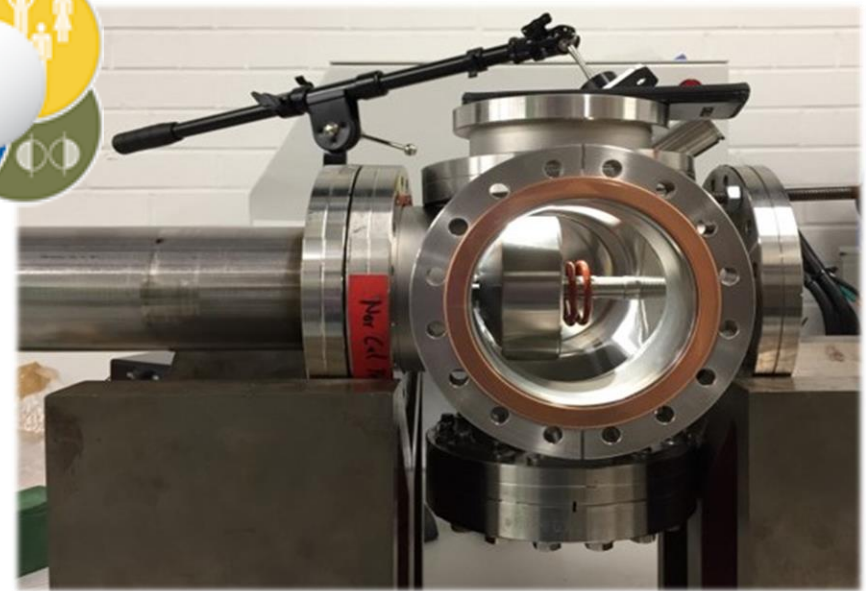
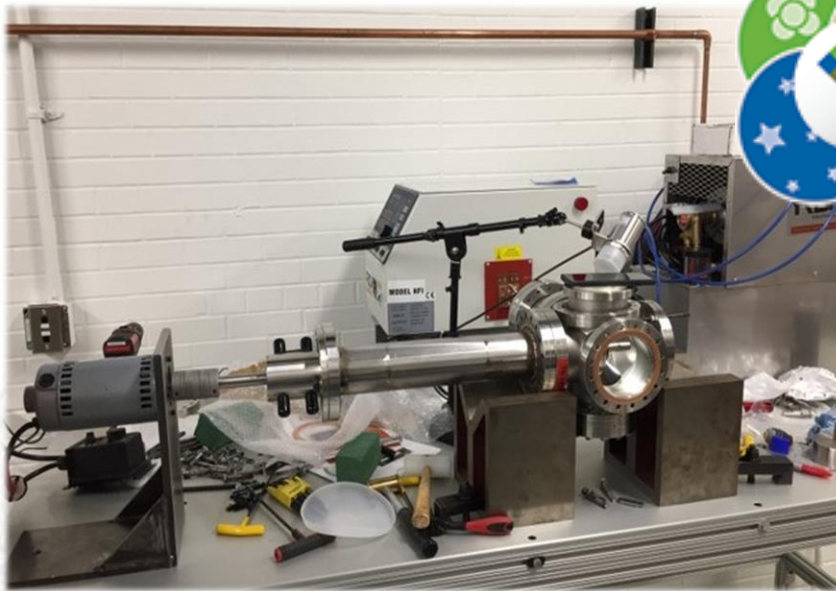
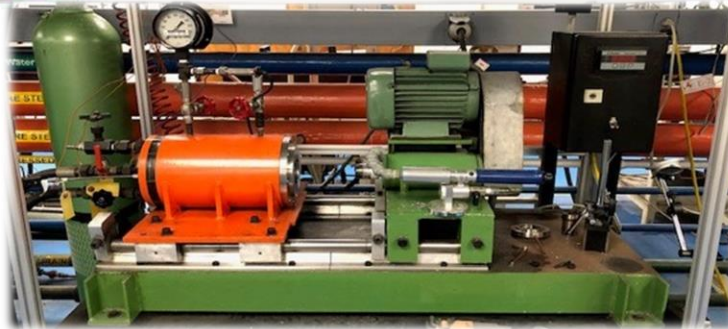
## for Delivery to MSU September 2022



1. The seal assembly shall isolate water at 27 PSIG nominal from oil at 0 PSIG nominal and isolate the oil from ambient vacuum at  $10^{-5}$  Torr
2. The rotating surface outer diameter is 4.500" Nominally with a 4.350" maximum axial length available
3. The rotational speed is not to exceed 700 RPM and temperature not to exceed 200° C
4. The seal must mate with existing oil flush geometry to minimum hardware modification required.

# Garlock Test and MSU FRIB Test Plan

*Deliver Final Material to Garlock for Introduction into Flood-Gard Housing*



Test Bench to Evaluate the Water Seal for the Beam Dump Assembly under Thermomechanical Environment Close to FRIB Operating Conditions



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**Dr. Jie Wei, Dr. Jian Gao, Dr. Frederique Pellemoine  
and Dr. Georg Bollen MSU FRIB**

**POCs: Dr. Michelle Shinn, Dr. Elizabeth Bartosz,  
Brenda May, John Motz, Christine Grady, Cassie  
Dukes, Linda Severs, Dr. Manouchehr Farkhondeh,  
and Dr. Manny Oliver**

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