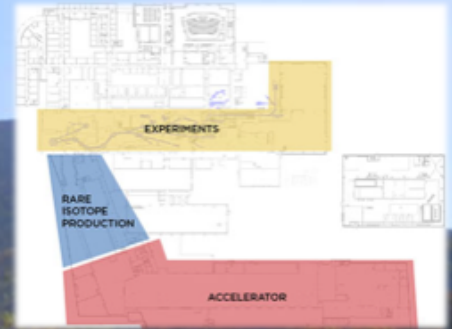




## ***Long-Term Radiation Rugged Rotary Vacuum and Water Seals in Heavy-Ion Accelerators***

NanoSonic, Inc.: Dr. Jennifer Lalli  
Colorado State University: Dr. Thomas Borak  
MSU FRIB: Dr. Frederique Pellemoine



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

August 14, 2019 - DOE SBIR Phase II DOE NP SBIR Exchange

POCs: Dr. Michelle Shinn, Dr. Elizabeth Bartosz, Mark Sojka, Moriam Vaughn, Cassie Dukes, Manouchehr Farkhondeh, and Mannie Oliver

# Overview

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

### Timeline:

- Project Start Date:  
*5/21/2018*
- Project End Date:  
*9/20/2020*

### Budget:

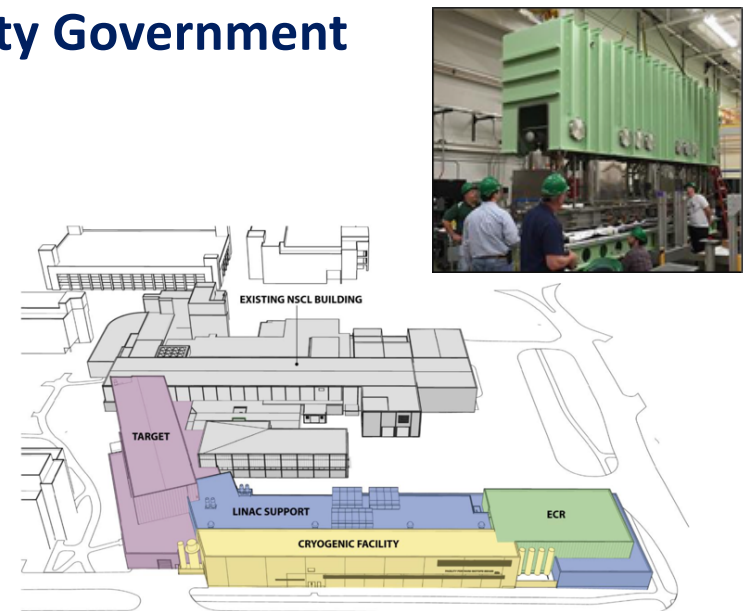
- FY17 DOE Ph I Funding:  
\$150,000
- FY18 DOE Ph II Funding:  
\$500,010
- FY19 DOE Ph II Funding:  
\$500,000
- Total DOE Project Value:  
\$1,160,000

### Needed for NP Experiments:

- Ultra-high vacuum and water cooled seals
- Under constant rotation at 600 rpm for ~ 1 year
- Extremely high radiation dose (100s of MGy)
- Need to change seal as infrequently as possible

### Partners:

- Giles County Government
- CSU
- MSU, FRIB
- Garlock
- BalSeal





# 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

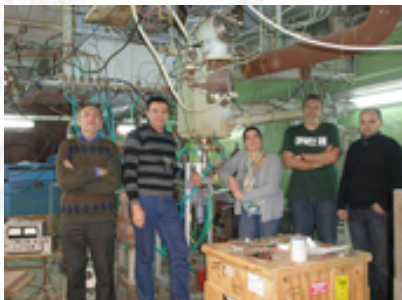
- >20 years of laboratory safety and production expertise
- Leads NanoSonic scale-up and product certification
- Commercializing Zero Humidity Fuel Cell Membranes with LANL



**Dr. Thomas Borak**, Radiation Physics and Safety Lead

Ph.D. Physics Vanderbilt

- Professor at CSU, Dept. of Environmental and Radiological Health Sciences
- Previous staff appointments at: Fermilab, CERN, and Argonne National Laboratory
- Distinguished Emeritus Member of the National Council on Radiation Protection (NCRP)
- Certified by the American Board of Health Physics



**Dr. Frederique Pellemoine**, MSU FRIB Seal Integrator

Ph.D. Experimental Physics Systems, University of Caen - France

- Staff Physicist, Target and Beam Dump Systems Group Leader (FRIB)
- International Nuclear Target Development Society (INTDS)
- Simulation Engineer, Grand Accelérateur National d'Ions Lourds (GANIL)
- Referee EMIR (network of accelerators dedicated to materials irradiation)



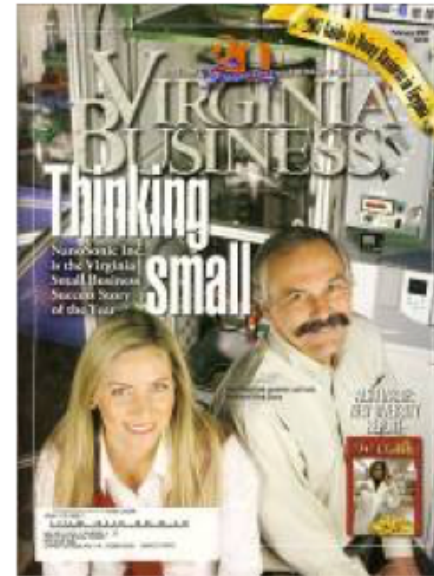
Facility for Rare Isotope Beams  
at Michigan State University

# NanoSonic Background

MARTINUS SCIENT  
**A LIMBER  
 FUTURE**  
 "Smart skin" holds promise  
 for morphing wings and  
 wearable computers.



- 1998 - Founded by Dr. Richard Claus Delaware C Corp., Blacksburg, VA
- Awarded Top Small Business in VA
- Top 5 Small Business at DARPA Tech
- Top 13 NASA Nanostructured Products
- 2004 – Alliance Agreement with LM
- 2007 – R&D 100 for Metal Rubber™
- 2008 - Micro/Nano 25 – Metal Rubber Fabric
- 2010 – New Manufacturing Facility
- 2011 - R&D 100 for HybridSil® Fire/Blast
- 2014 - Sikorsky Innovation Award



## Lockheed Martin and NanoSonic, Inc. sign partnership to study nanotechnology applications

Bethesda, MD, July 28, 2004— Lockheed Martin Corporation (NYSE: LMT) and NanoSonic, Inc., today signed a multi-year agreement to work on general market developments related to products, services, customer implementations and current projects related to the field of nanotechnology.

This agreement allows both companies to collaborate on emerging nanotechnology initiatives with many applications expected in the aerospace and defense industries. Technical advances to be pursued include those in the nanosensory and nano-coating areas. Both parties expect to provide improved technical, price and delivery considerations to their customers.

Dr. Sharon Smith, Lockheed Martin director of technology said, "There are a number of excellent nanotechnology companies debating around the country, and we feel that NanoSonic's research and development efforts align with some of the needs we currently have at Lockheed Martin. We look forward to a prosperous alliance."

"We believe our partnership with Lockheed Martin will prove to be very beneficial for our Blacksburg, Va., company. And it will boost Virginia's efforts in the relatively new field of nanotechnology," said Dr. Richard O. Claus, president of NanoSonic and a chaired professor of chemical and computer engineering and materials science at Virginia Tech.

Both Claus and Smith are new appointees to Virginia's Joint Commission on Technology and Sciences' Advisory Committee on Nanotechnology.

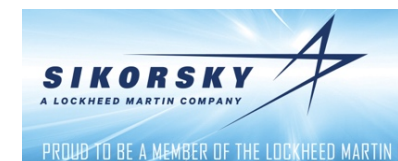
NanoSonic, headquartered in Blacksburg, Va., was created in 1998 in cooperation with Virginia Tech, and the State of Virginia. It recently announced its manufacturing of a new material called METAL RUBBER™ that conducts electricity like a metal, but stretches like rubber to up to several hundred percent of its original length. The material is being investigated for varied uses from the biomedical to the aerospace industries.

Headquartered in Bethesda, Md., Lockheed Martin employs about 130,000 people worldwide and is principally engaged in the research, design, development, manufacture and integration of advanced technology systems, products and services. The corporation reported 2003 sales of \$31.8 billion.

Contact:

[www.nanosonic.com](http://www.nanosonic.com)

540-426-4206



# Our Manufacturing Facility Timeline

10,000 sf beginning, 30,000 sf today, planned 100,000 sf production site



...yesterday



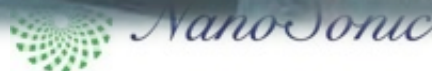
...today...



tomorrow...



Year	Facility	Production Volume
2000	10,000 sf	100 L / day
2010	30,000 sf	255 gal / day
2020	130,000 sf	> 2,000 gal / day



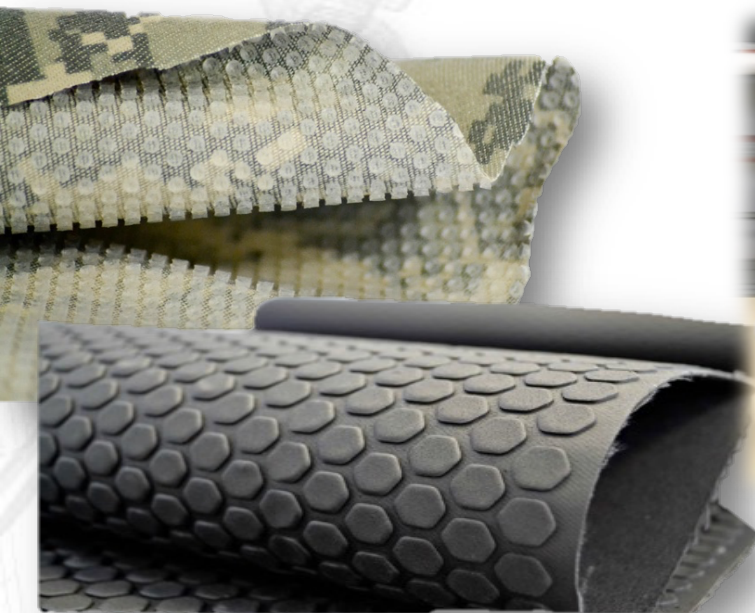


# Our Commercial Products

## 15 SBIR Derived on Webstore



[www.nanosonic.com](http://www.nanosonic.com)



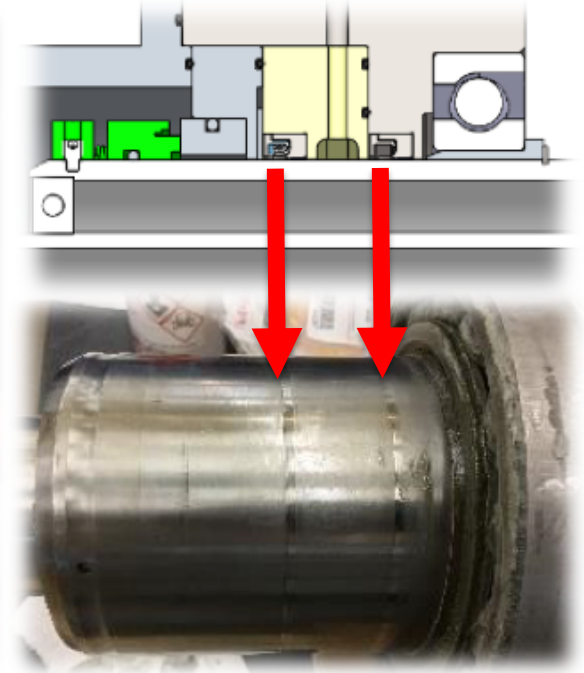
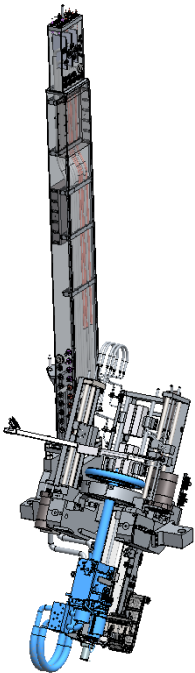
NanoSonic

# 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 exposed to 300 - 600 MGy minimum 1 year (6,000 hours) rotating at 600 rpm over 32 °C to 66 °C, water side: 60 gpm (25 psi), vacuum side:  $1e^{-5}$  Torr L/s



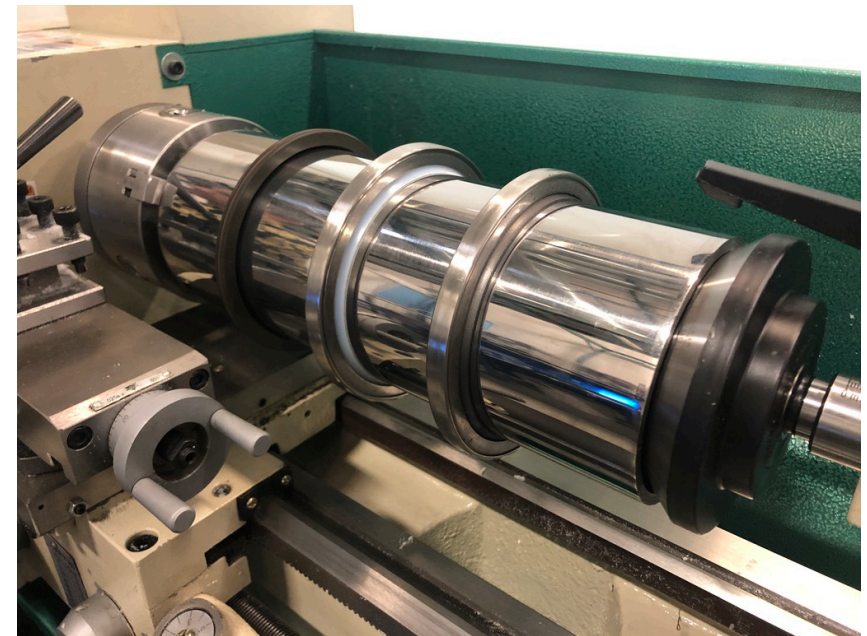
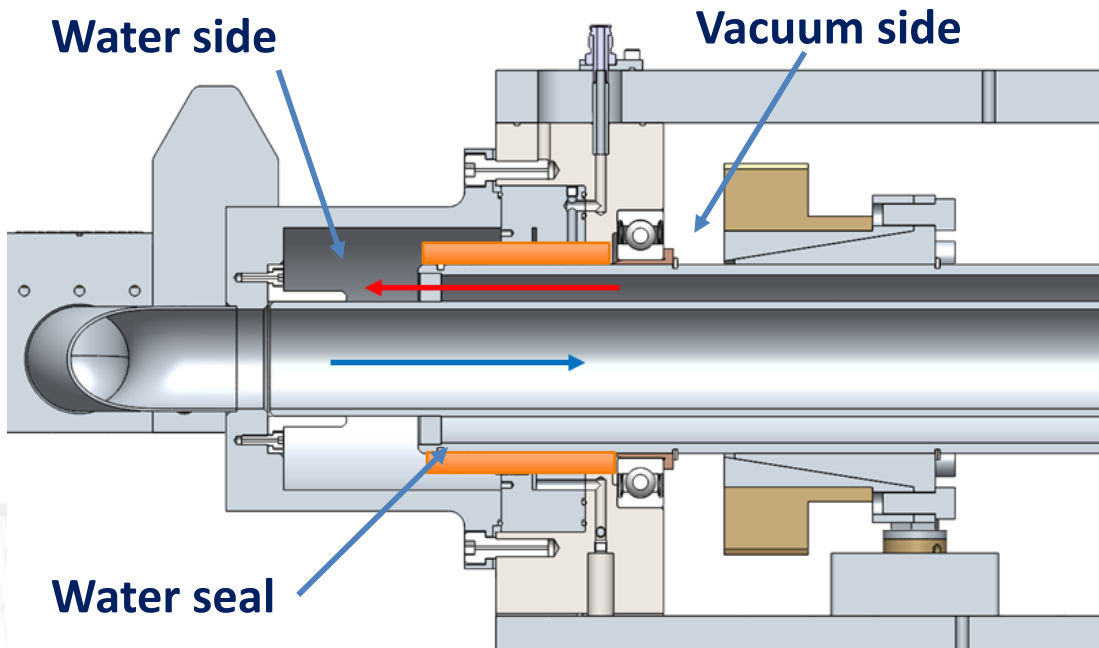
*Need the mechanical performance of Teflon with enhanced Radiation & Abrasion Resistance  
May need new material and new design*

# Relevance:

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

### GOALS:

- Developing new Teflon like, non-fluorinated materials with enhanced radiation resistance
- Utilizing extrusion for compounding new films not previously obtainable
- Investigating new seal design – Flood-Gard™ as opposed to traditional lip seal



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

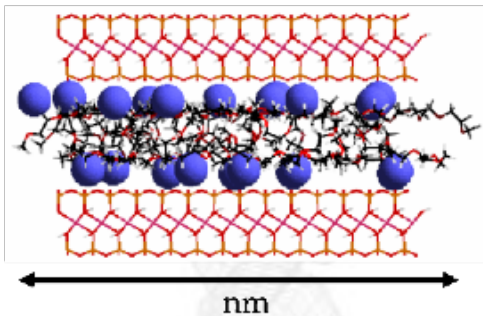


# Technical Approach

*Extrude New Compounded Materials for use in New Lip and Flood-Gard Seals*

## 1. Design New Materials

- For current cores
- Address leakage
- Custom OD and ID



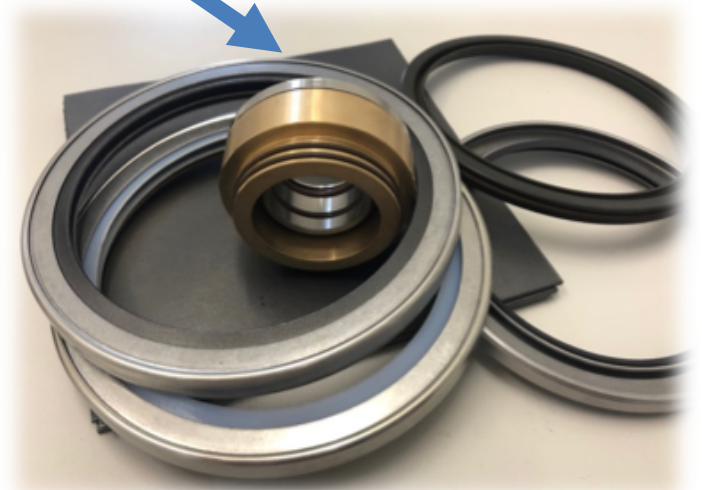
## 2. Design New Seals

- For new fittings
- Advanced materials
- Custom ID



**NEW SEAL**  
Opposing lips  
for  
Water and Vacuum

Stationary seal  
for  
No rotation on drum



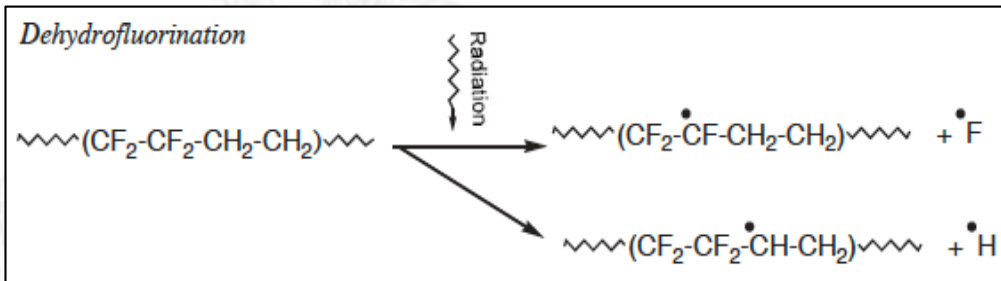
# Current Seals

## Material and Design Drawbacks

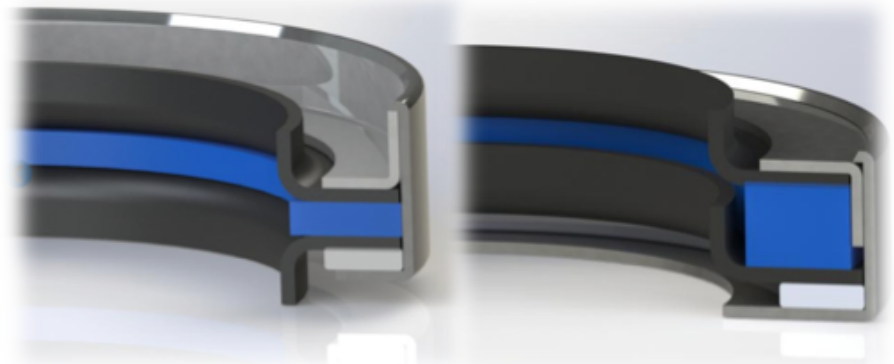
### 1. Material – Dehydrofluorination

A major drawback of Tefzel is that fluorinated polymers, particularly those containing hydrogen, evolve hydrogen fluoride (HF) upon irradiation. Higher hydrogen contents increase the tendency for dehydrofluorination:

PTFE > PFA > ~ FEP > **ETFE** > PVDF > PVF



### 2. Seal – Need Opposing Lips



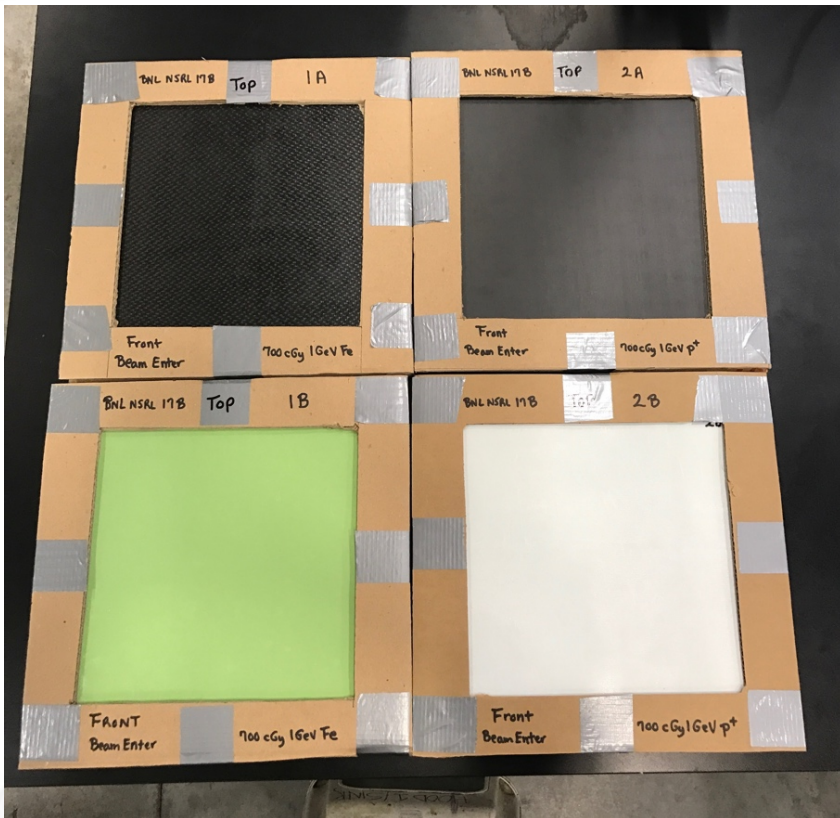
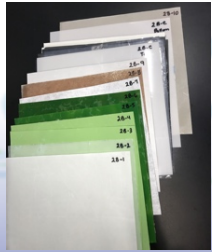
Or new design



*HF is typically carried off in vacuum,  
may or may not be an issue for water in water cooled seals*

# Accomplishments in Irradiation

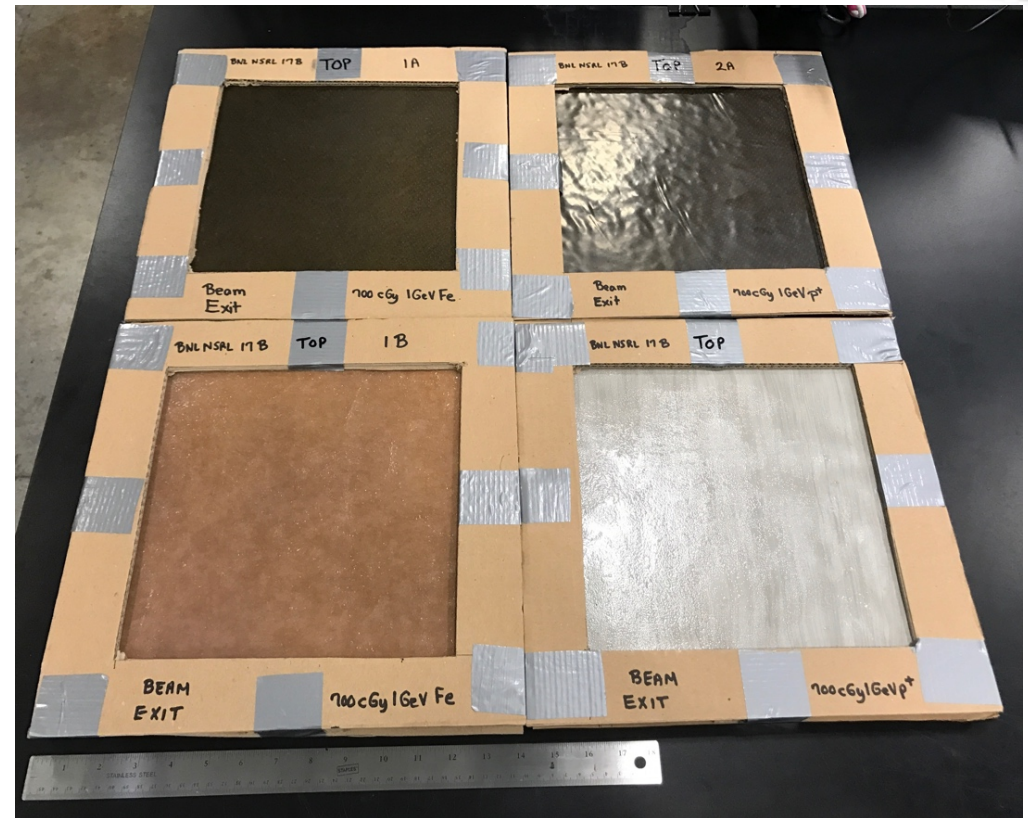
*Exposure to Fe and proton at BNL NSRL*



**Front – Beam Enter View**

**2 Cassettes for Fe – 1 GeV**

**2 Cassettes for proton – 1 GeV**



**Back – Beam Exit View**

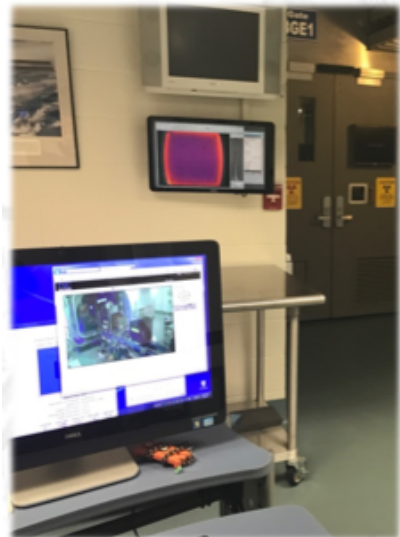
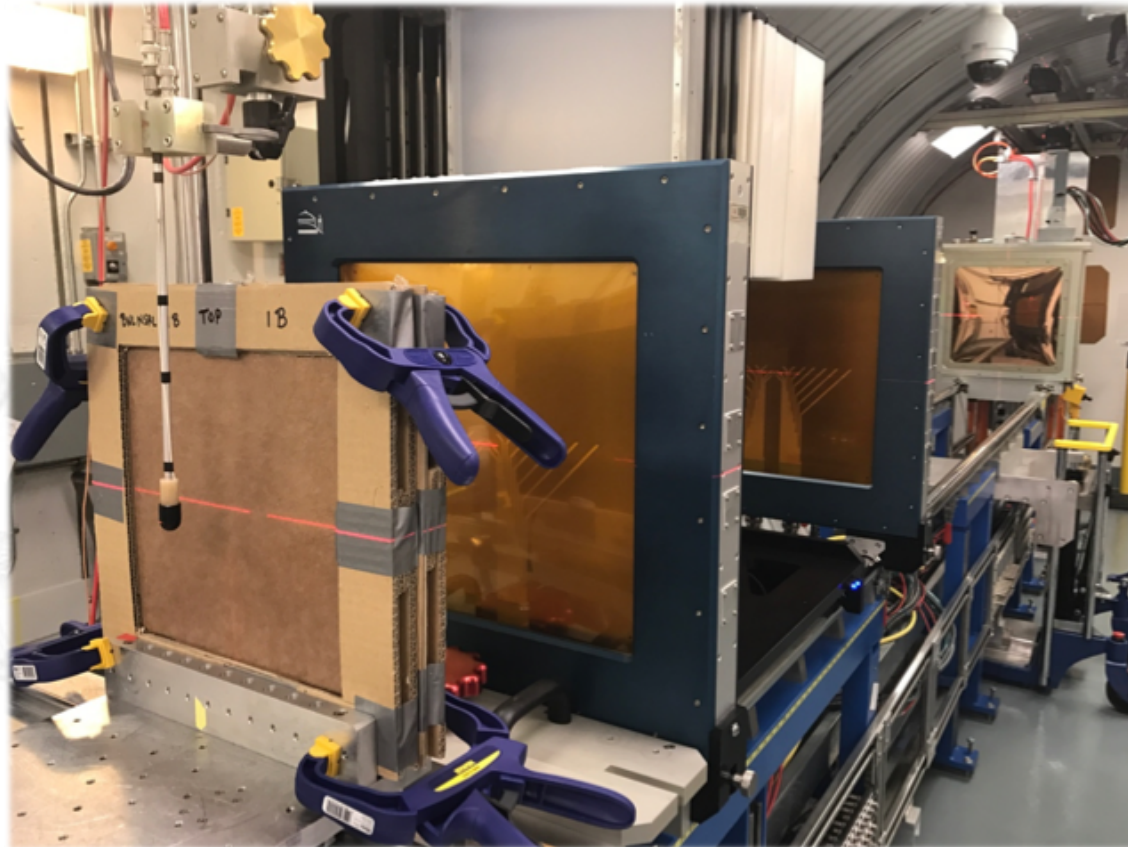
**2 Cassettes for Fe – 1 GeV**

**2 Cassettes for proton – 1 GeV**

***4 Cassettes Prepared for Fe and proton irradiation – 27 Gy***

# Accomplishments in Irradiation

*BNL NSRL Run During Phase I*



***PI is Rad 1 Worker Certified at NSRL  
Renewal for 2 years Completed June 2019***



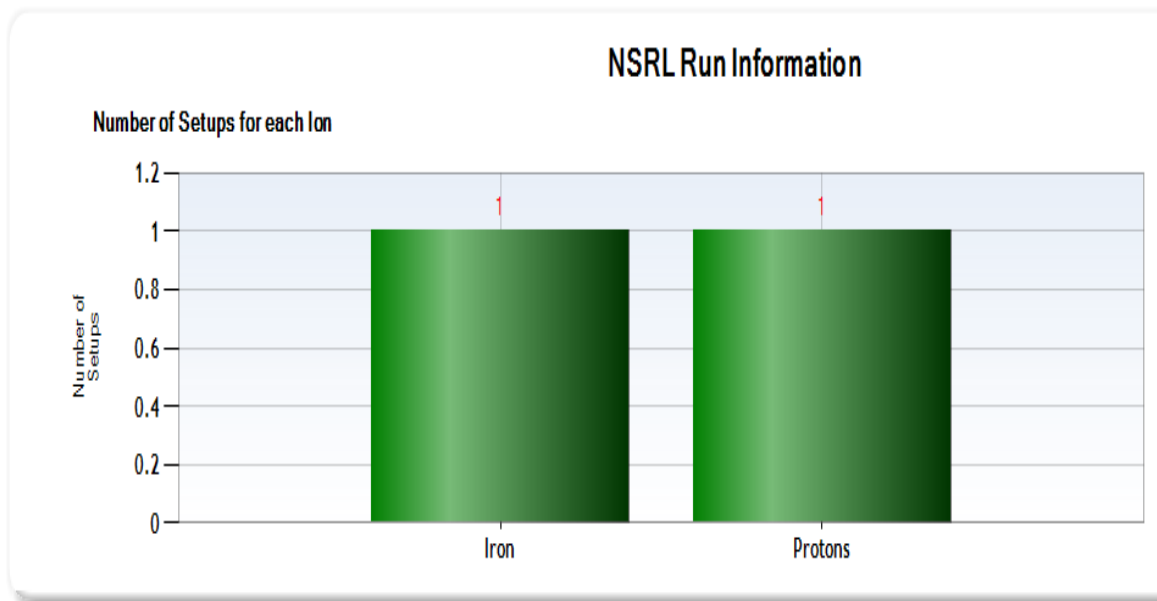
# Accomplishments in Irradiation at BNL NSRL

June 8, 2017

Displaying records 1 thru 2 of 2 record

First Previous 1 Next Last

Run Number	Operator Name	Ion	Energy (MeV/n)	NASA Approved Hrs.	Work Type	Dose Rate (cGy/min)	Dose Range (cGy)	Fluence (particles/cm <sup>2</sup> )	Particles Per Spill (particles/cm <sup>2</sup> /spill)	Number of Samples	Start Setup	Start Exposure	Start Wrap-up	Run Time
17B	Rosselot, Rory	Protons	1000	1.58	Physics	1	1			1	6/8/2017 12:02:26 PM	6/8/2017 12:16:45 PM	6/8/2017 12:36:52 PM	00:20:07
17B	Rosselot, Rory	Iron	1000	1.58	Physics	1	1			1	6/8/2017 10:36:31 AM	6/8/2017 10:36:31 AM		01:25:55



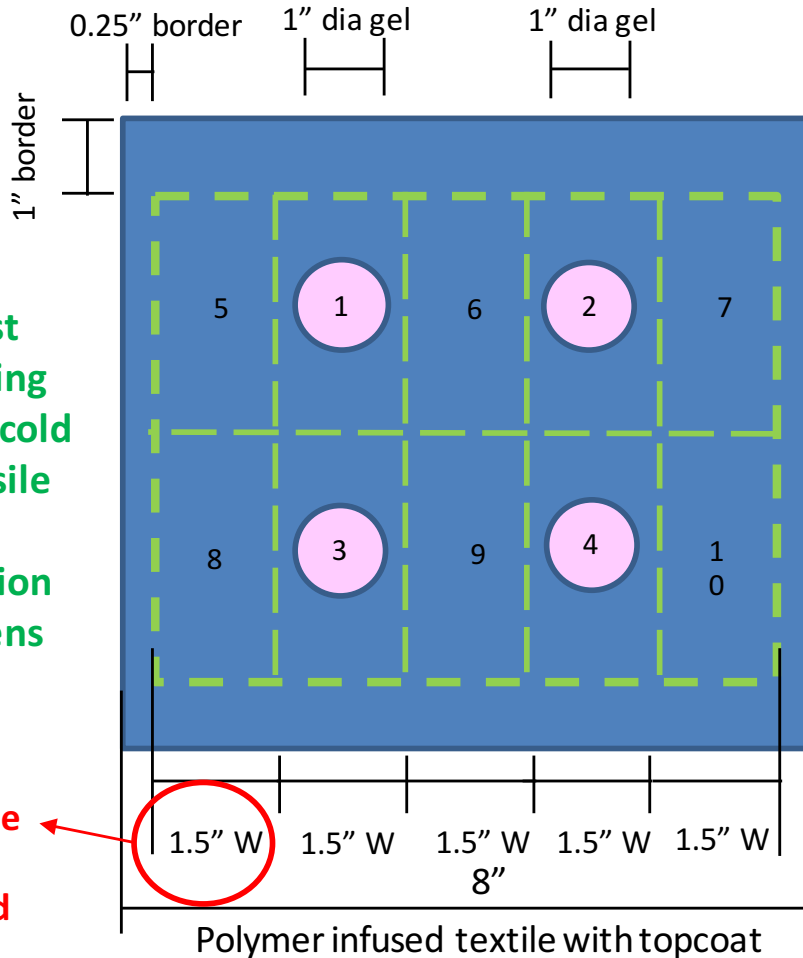
*Fe and proton irradiation – 27 Gy*

# Accomplishments in Radiation Durability Studies

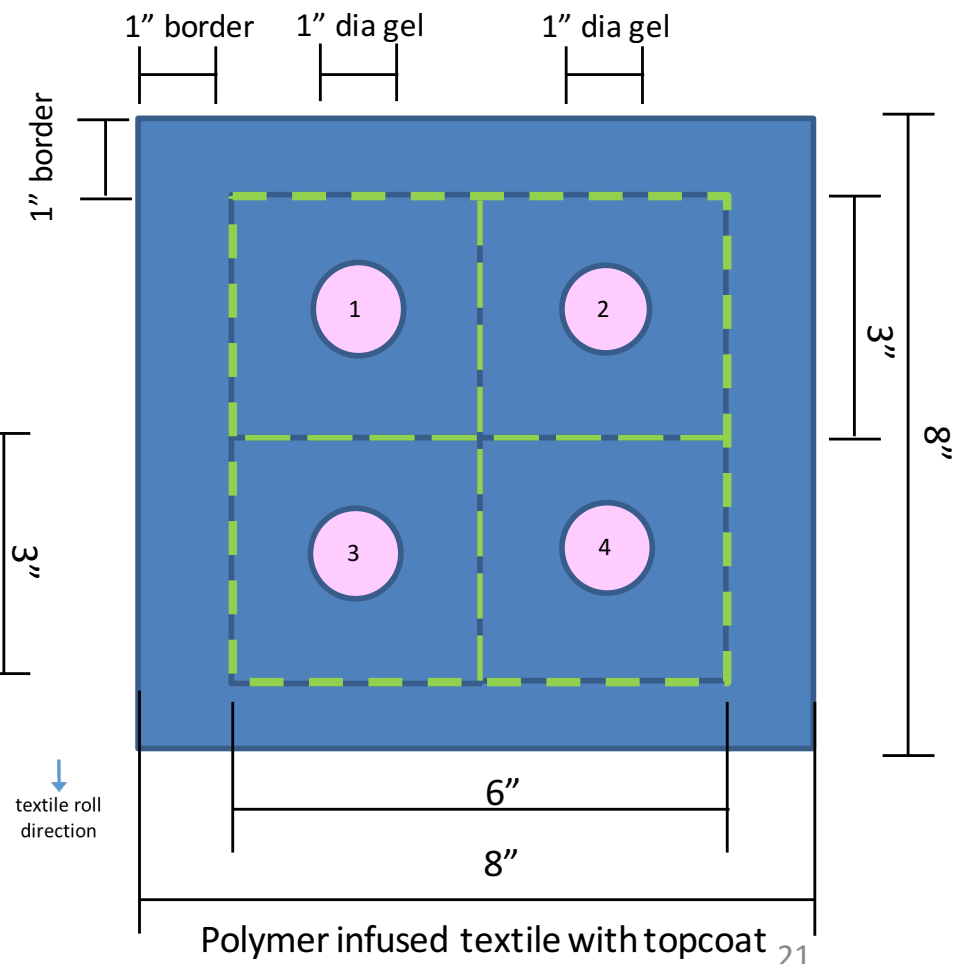
Evaluate abrasion and thermo-mechanical properties post irradiation

RUN 1B  
RUN 6B  
with gel

four 1.5x3" cold flex/tensile specimens w/ gel (#1,2,3,4)  
+ six 1.5x3" w/o gel (#5,6,7,8,9,10) per layer



four 3x3" permeation specimens (1,2,3,4) per layer

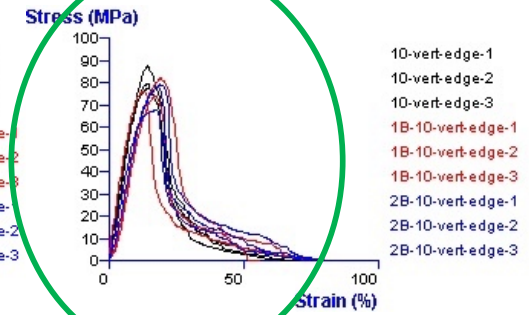
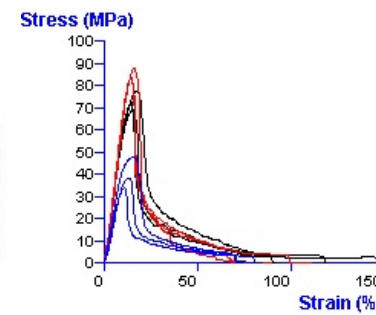
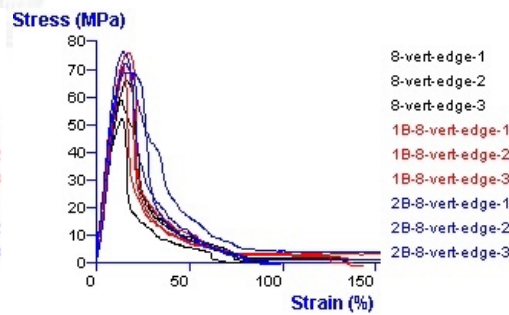
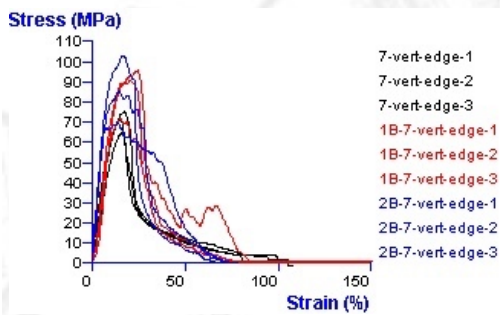
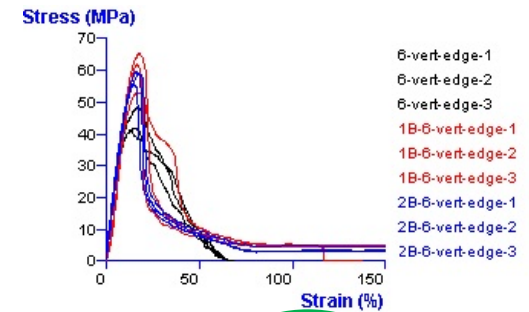
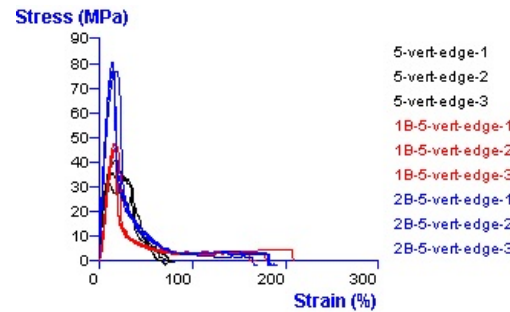
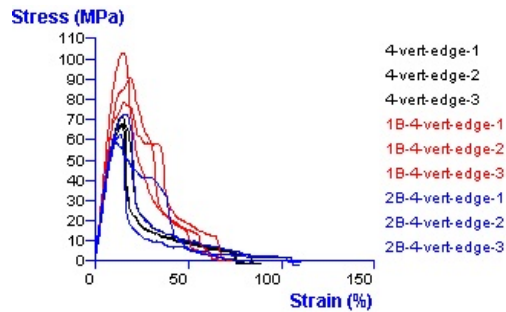
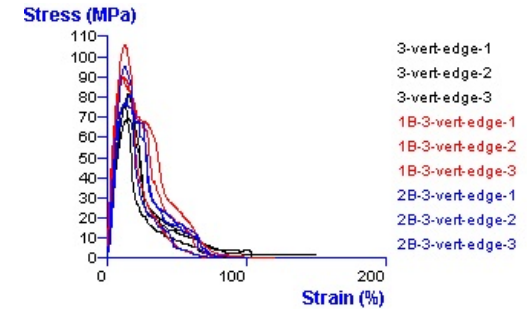
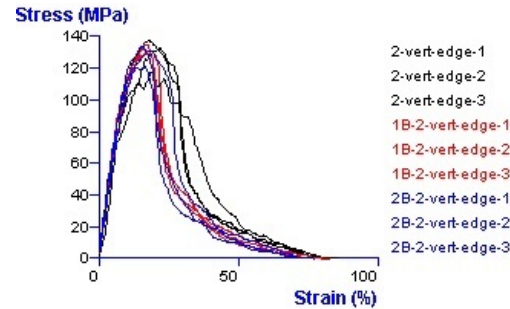
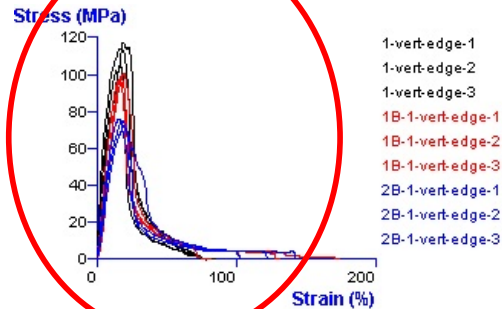
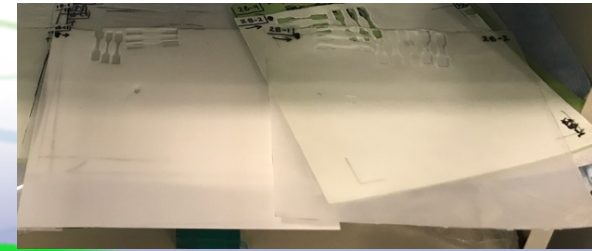


Suggest alternating layers for cold flex/tensile and permeation specimens

May use 1x3"s instead

Exemplary Cutting Patterns Post Radiation Required for Tensile, DMA, and Abrasion

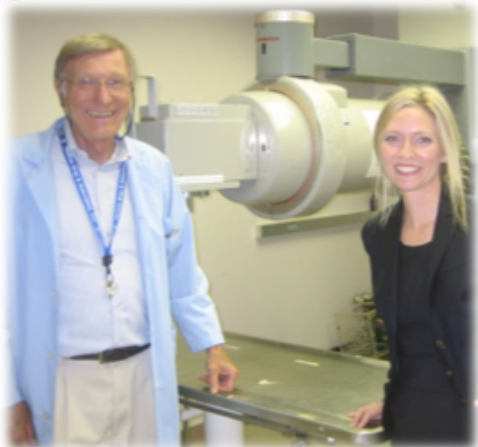
# NanoSonic Vertical Pre- and Post- Irradiation



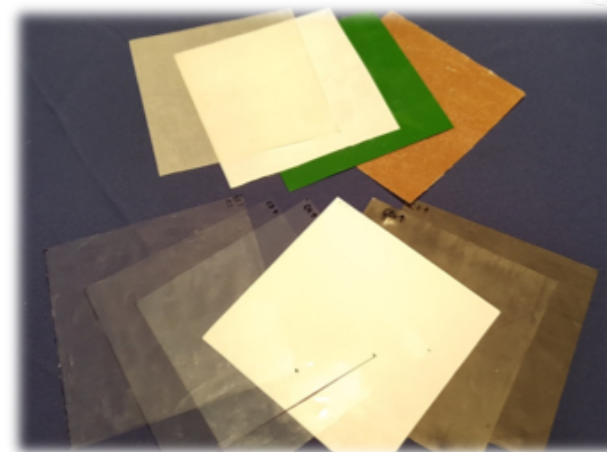
Note Tensile Strength Variations or Stability Post **Fe** and Proton Exposure

# Accomplishments in electron Irradiation

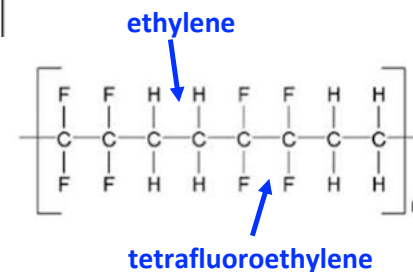
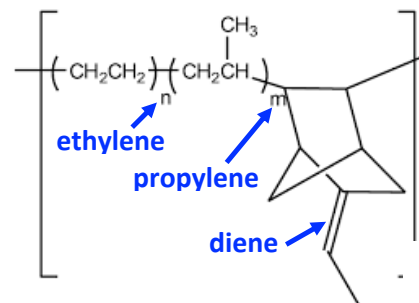
## *electron Irradiation – 27 Gy at CSU*



**Varian Trilogy™ Linear Accelerator with, 6 and 10 MV photons and 4,6,9,12 and 15 MeV electrons**



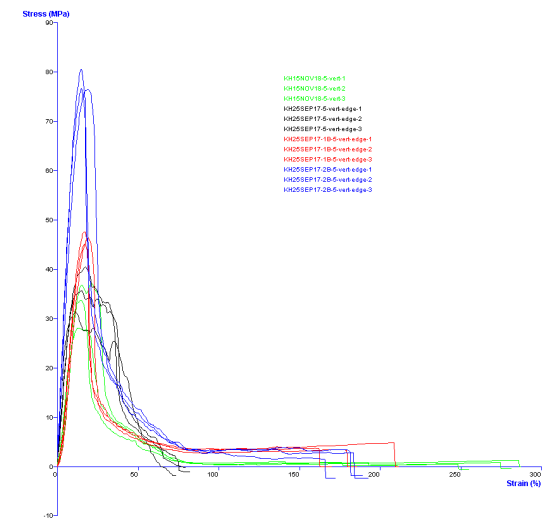
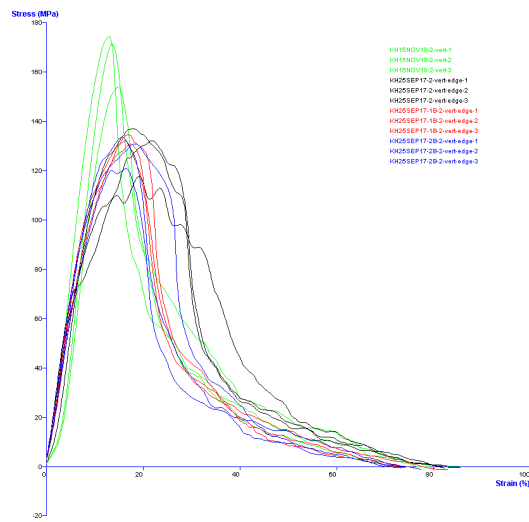
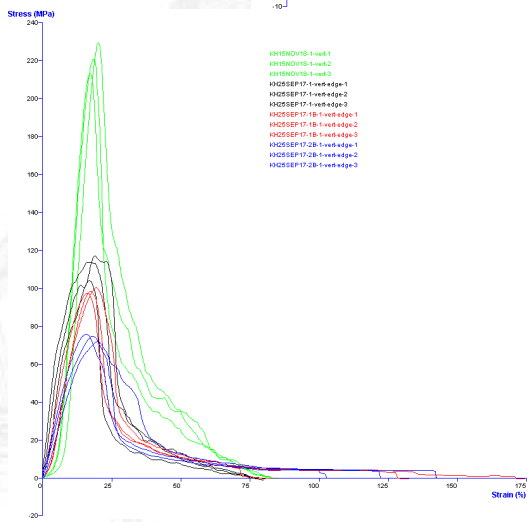
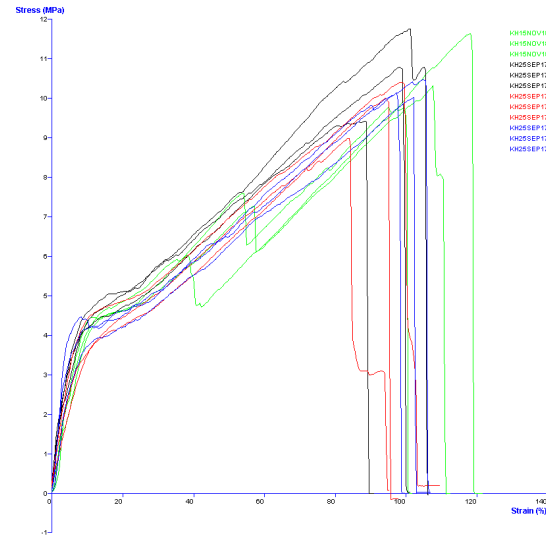
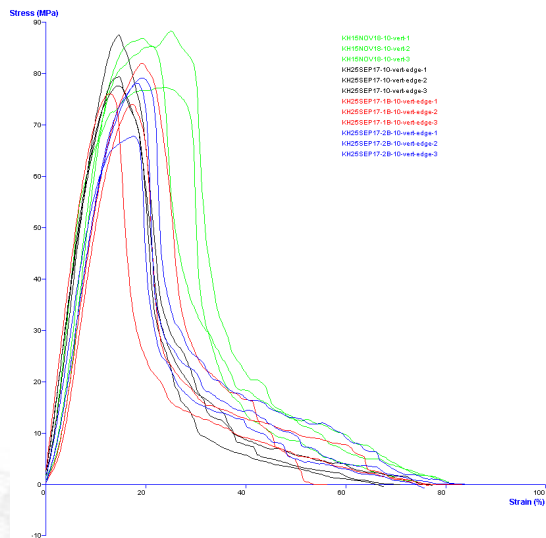
Description	Sample ID	Length (cm)	Width (cm)	Weight (g)	Areal Density (g/cm <sup>2</sup> )	Thickness (cm)
NanoSonic Materials	1	20	20	12.9	0.03225	0.285
	2	20	20	15.3	0.03825	
	5	20	20	42.8	0.107	
	10	20	20	27.2	0.068	
Commercial Materials	C2	20	20	2	0.005	0.176
	C3	20	20	4	0.01	
	C4	20	20	9	0.0225	
	C5	20	20	43.7	0.10925	
	C6	20	20	14.5	0.03625	
	C7	20	20	16.5	0.04125	
					<b>0.47</b>	<b>0.461</b>
					<b>Total Areal Density</b>	<b>Total Thickness</b>



***Down-Selected Materials Exposed to 27 Gy electron at CSU***



# Accomplishments in Mechanical Properties Post Low Dose Exposure Electron, Fe, and Proton Irradiation – 27 Gy

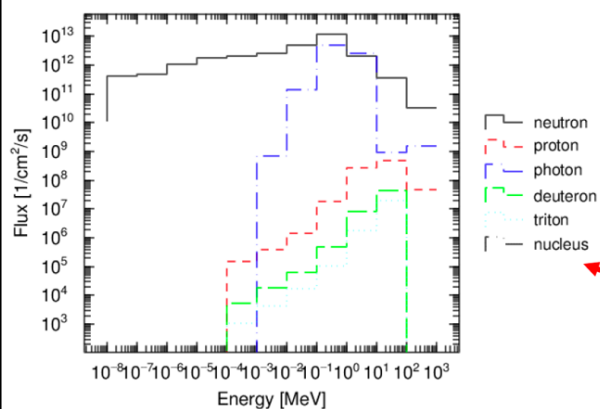


**Tensile Strength Stability Post Fe, proton, and electron Exposure in both Directions**

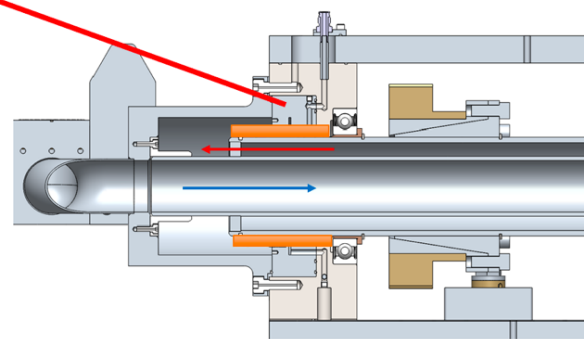
# Accomplishments in High Dose Exposure

## Plans for 600 MGy electron, proton, and Fe

■  $^{48}\text{Ca}$  at 400 kW, 261 MeV/u  $\rightarrow$  40Mg



D. Georgobiani



Energy, MeV		Flux, particles/cm2/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

We will expose candidate seal materials to electron irradiation using high dose medical sterilization techniques at 50 MGy per pass for 100, 200, and 300 MGy doses at Steris – and BLIP



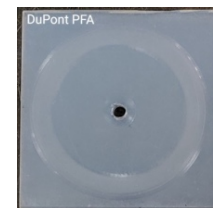
**Will Conduct 600 MGy electron exposure at Steris and 600 MGy Fe, proton at BNL's LINAC**

# Accomplishments in Abrasion

## On Taber Abrader per ASTM D-1003



Taber Abrasion for Water/Vacuum Seals						
CS17 Diameter						
Material	L (mm)	R (mm)	Pretest weight (g)	Post-test weight (g)	cycles	Loss (g)
Tefzel 3mil	51.19	50.88	1.0513	1.0408	4	0.0105
Tefzel 5mil	51.06	50.68	2.4301	2.407	1000	0.0231
Tefzel .093"	50.94	50.62	47.1957	47.1734	1000	0.0223
Daikin PFA	48.78	47.84	101.8792	101.8357	1000	0.0435
Dupont PFA	48.69	47.81	101.4681	101.4392	1000	0.0289
McMaster PTFE	48.59	47.79	53.83	53.44	1000	0.3900
C-Plastics PTFE	48.49	47.58	55.9317	55.6309	1000	0.3008

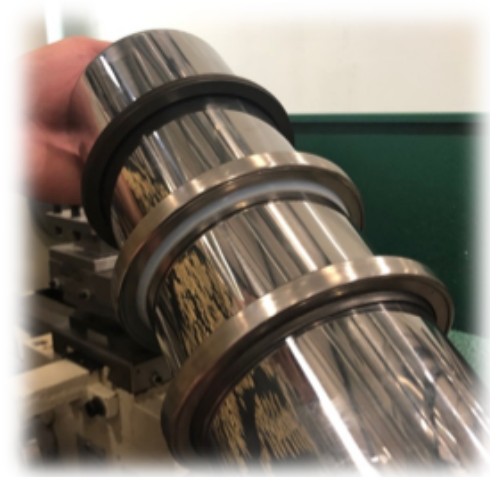
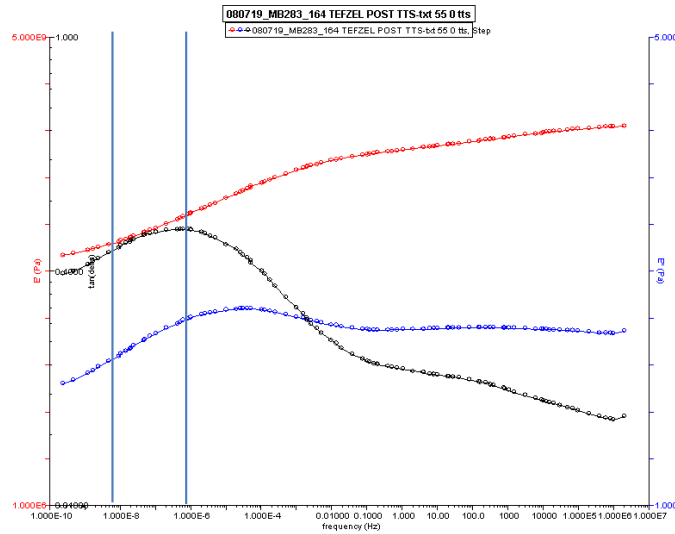
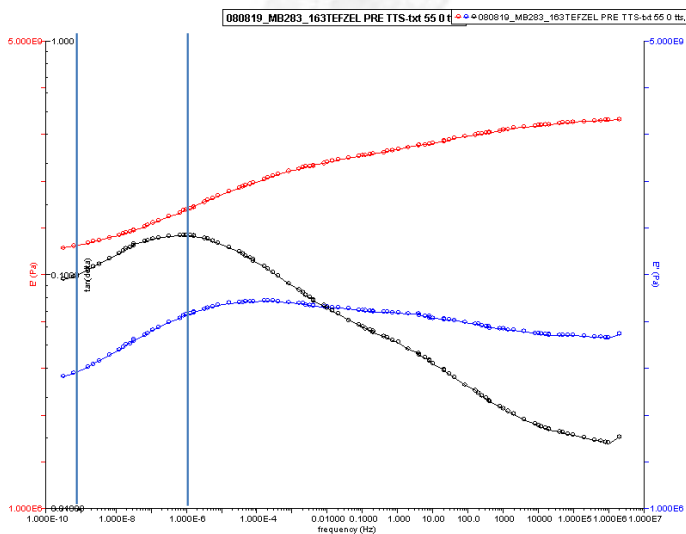
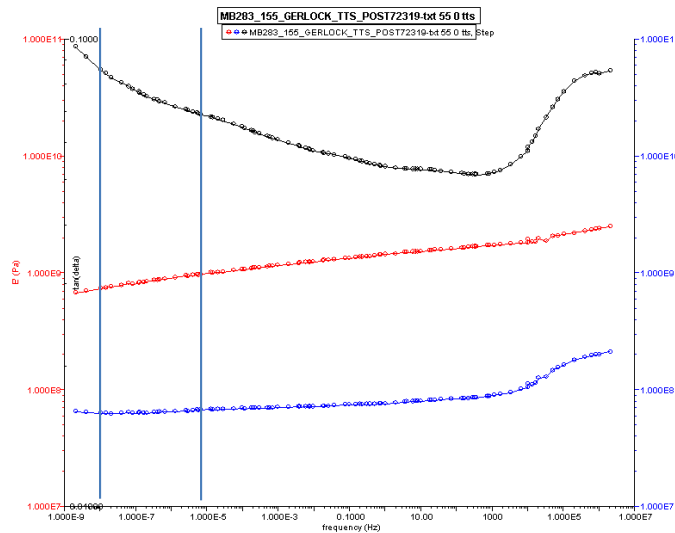
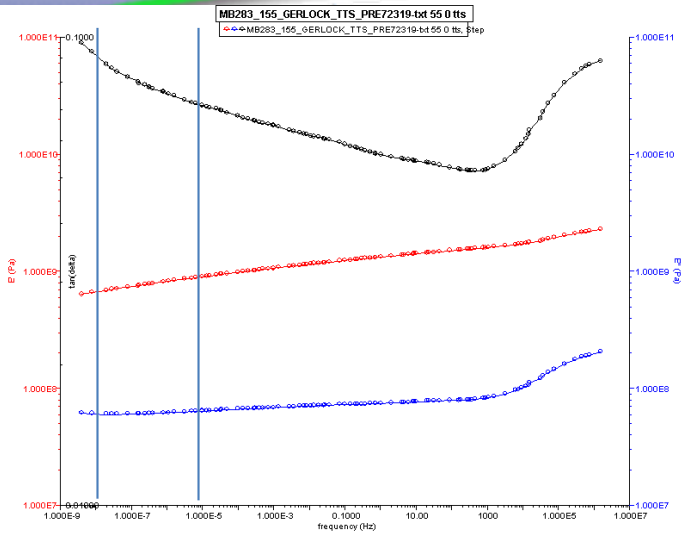


***Tefzel offers lower weight loss relative to Teflon as expected***  
***Rockwell Hardness of 50 is Important as SS 304I is 30 vs. SS 304 of 70***



# Accomplishments in Lifetime Assessment

## DMA TSS for 1-2 year predictive analysis



**Temperature Step Frequency Sweep Runs Allow for Tan Delta and Loss Modulus Predictions at Instantaneous Events or out to > 2 Years**

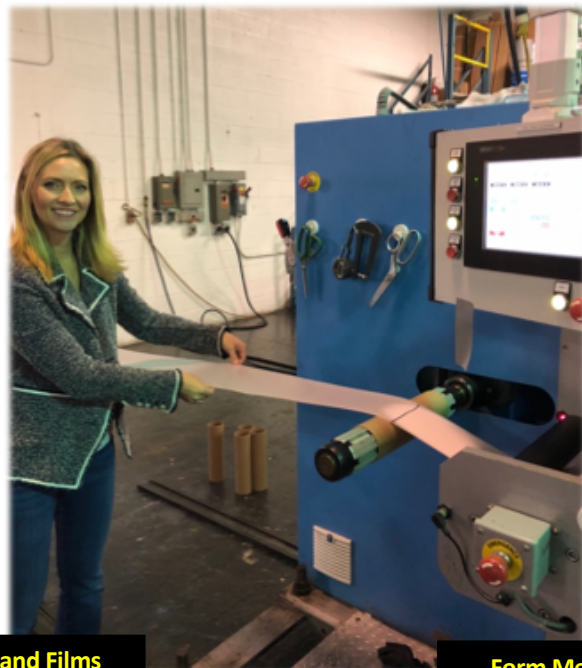
# Remaining Challenges and Barriers:

## *High Dose Testing and Final Seal Integration*

- Challenge: Testing post 600 MGy exposed materials in water seal target
- Resolution: Possible testing at PNNL if needed – perhaps in Ph IIA
- Challenge: Getting new materials integrated within seal
- Resolution: Partnered with Garlock for new seal design and Techsburg on machining



Leistritz Extruder and Films



Form Metal Housing for New Seals



***NanoSonic is Producing New Films for Use in Commercial Lip and Flood-Gard Seals***

# Acknowledgements

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Drs. Michelle Shinn and Elizabeth Bartosz, DOE**



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