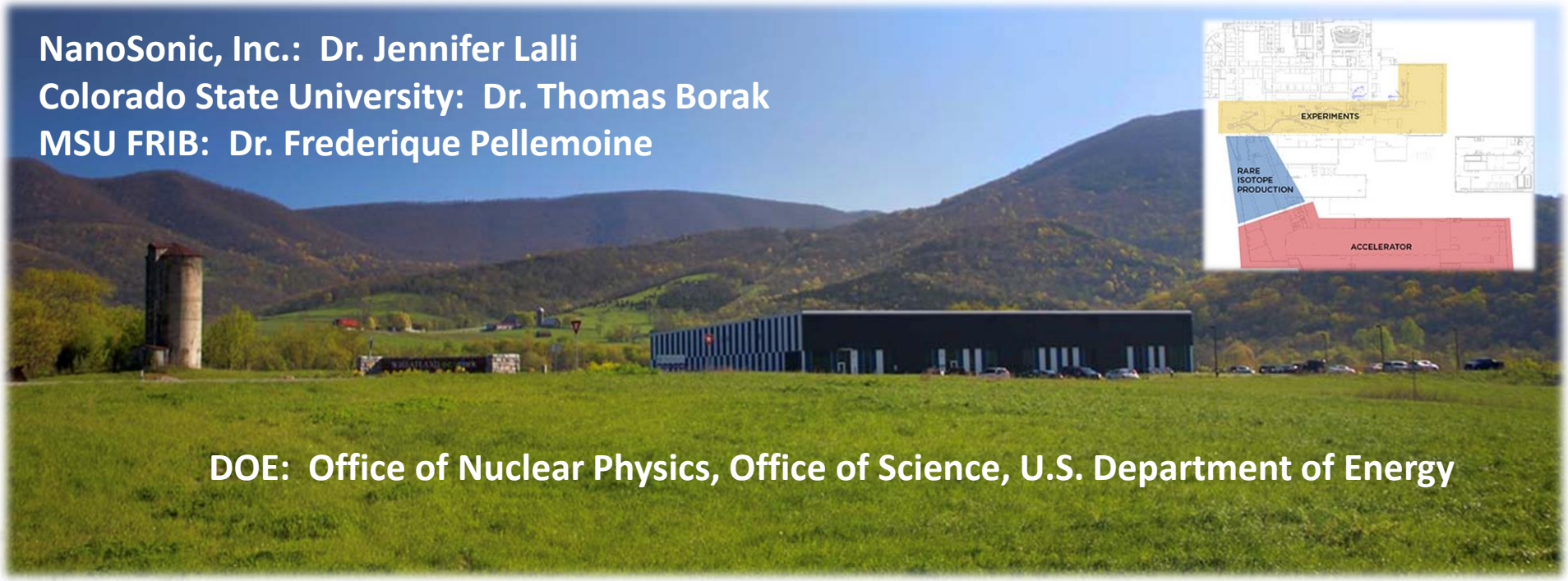
NanoSonic, Inc.

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, 2020 (*draft*) - DOE SBIR Phase II DOE NP SBIR Exchange
PROPRIETARY DRAFT

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*

Needed for NP Experiments:

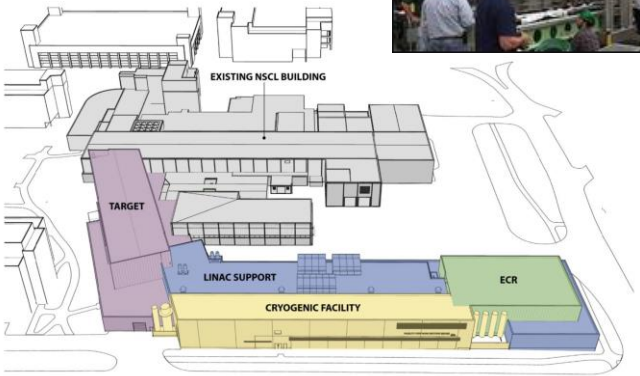
- Ultra-high vacuum and water cooled seals
- Under constant rotation at 600 rpm for ~ 1 year
- Extremely high radiation dose (100s of MGy)
- Need to change seal as infrequently as possible

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

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

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. Jian Gao,
Ph.D. Physics Vanderbilt

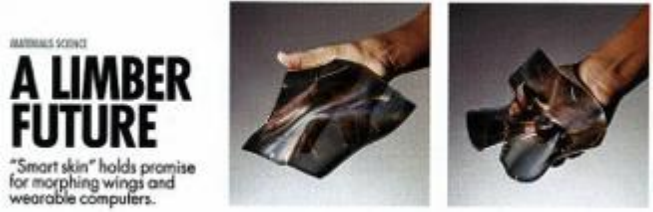
Target and Beam Dump Systems Group Leader
Facility for Rare Isotope Beams
Michigan State University

Dr. Frederique Pellemoine, MSU FRIB Seal Integrator
Ph.D. Experimental Physics Systems, University of Caen - France

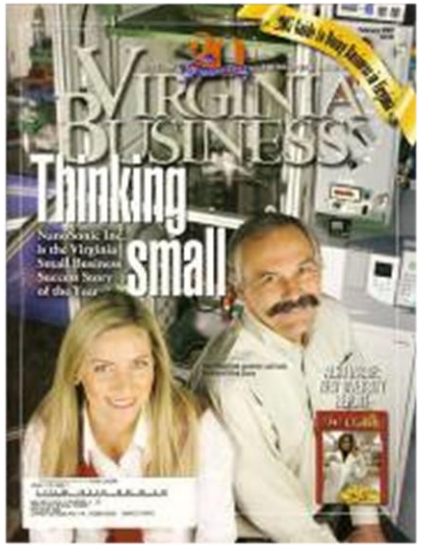
- Staff Physicist, Target and Beam Dump Systems Group Leader (FRIB)
- International Nuclear Target Development Society (INTDS)
- Simulation Engineer, Grand Accelerateur National d'Ions Lourds (GANIL)
- Referee EMIR (network of accelerators dedicated to materials irradiation)

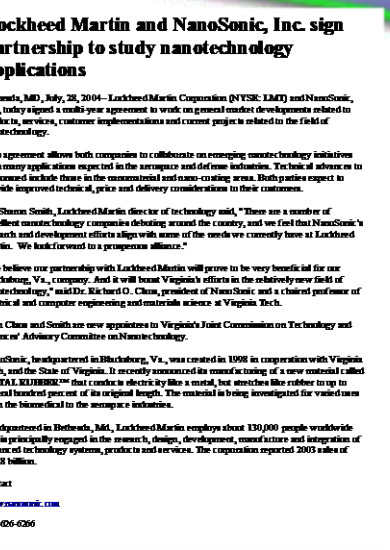
Facility for Rare Isotope Beams
at Michigan State

NanoSonic Background



- 1998 - Founded by Dr. Richard Claus Delaware C Corp., Blacksburg, VA
- Awarded Top Small Business in VA
- Top 5 Small Business at DARPATech
- 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 nanomaterial 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 debuting 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 electrical and computer engineering and materials science at Virginia Tech.

Ruth Claus and Smith are new appointees to Virginia's Joint Commission on Technology and Science' 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 in 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

540-026-6266

NanoSonic

Our Manufacturing Facility Timeline

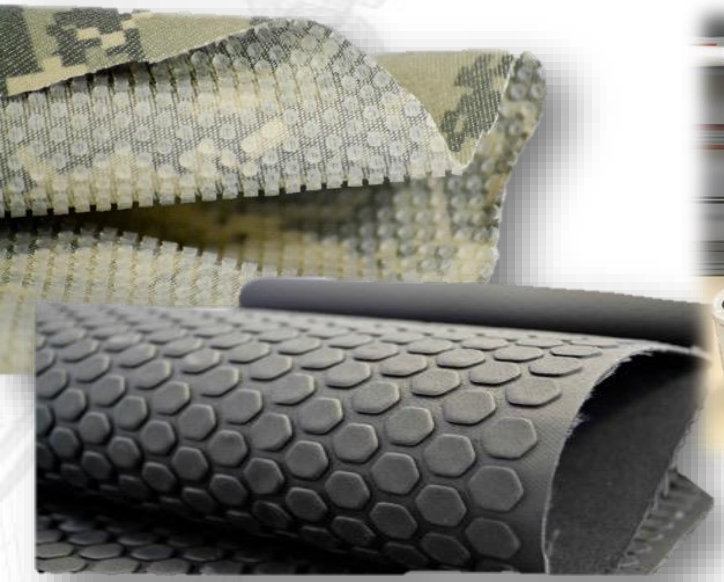
10,000 sf beginning, 30,000 sf today, planned 100,000 sf production site

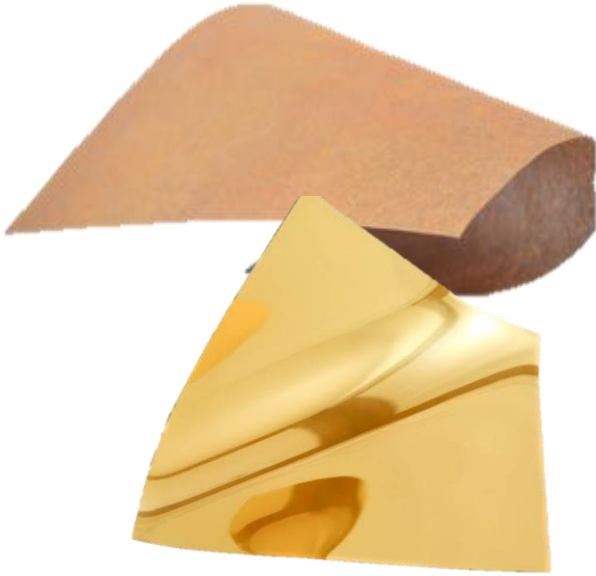
Year -	Facility	-	Production Volume
2000 –	10,000 sf	-	100 L / day
2010 –	30,000 sf	-	255 gal / day
2020 –	130,000 sf	-	> 2,000 gal / day

Our Commercial Products

15 SBIR Derived on Webstore

www.nanosonic.com





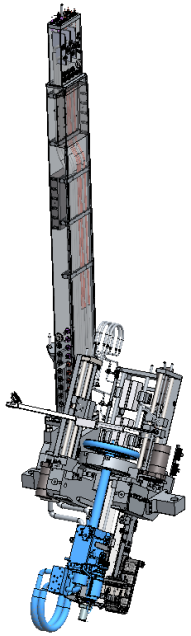
NanoSonic

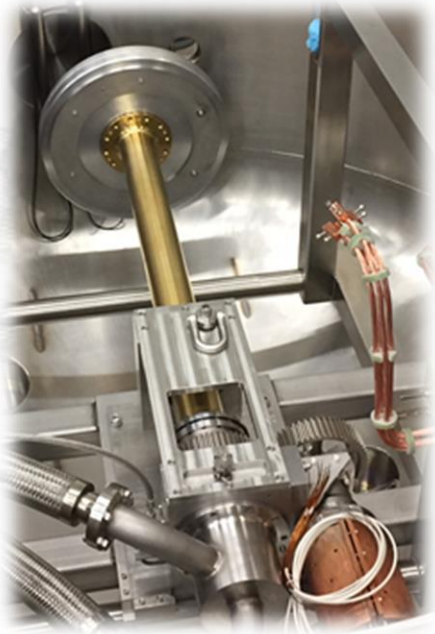
DOE Topic 26f – Technology for High Radiation Environments

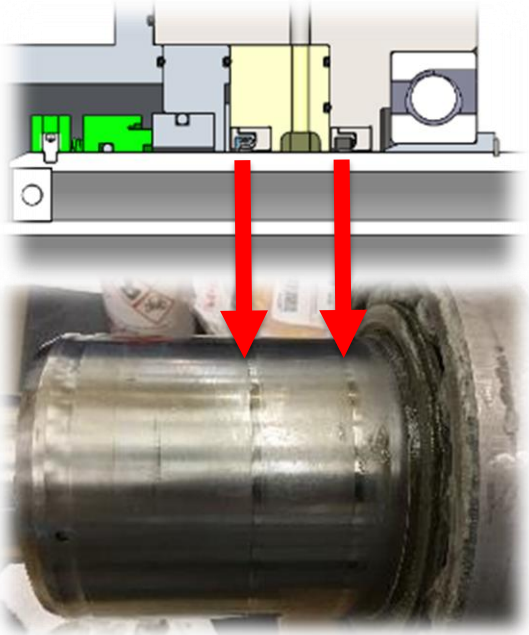
Grant # DE-SC0017107

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**







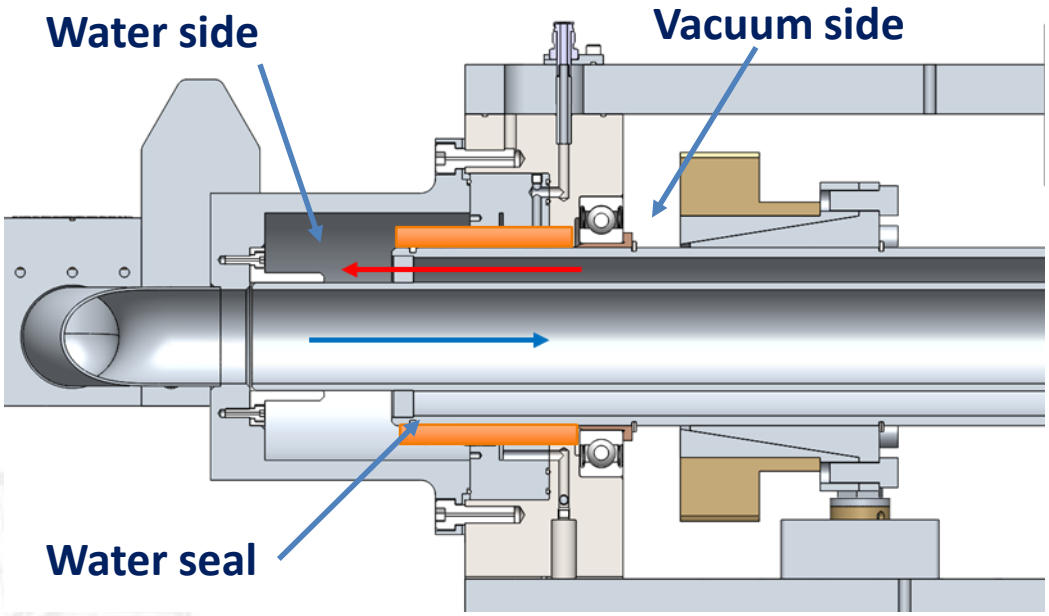
Need the mechanical performance of Teflon with enhanced Radiation & Abrasion Resistance
May need new material and new design

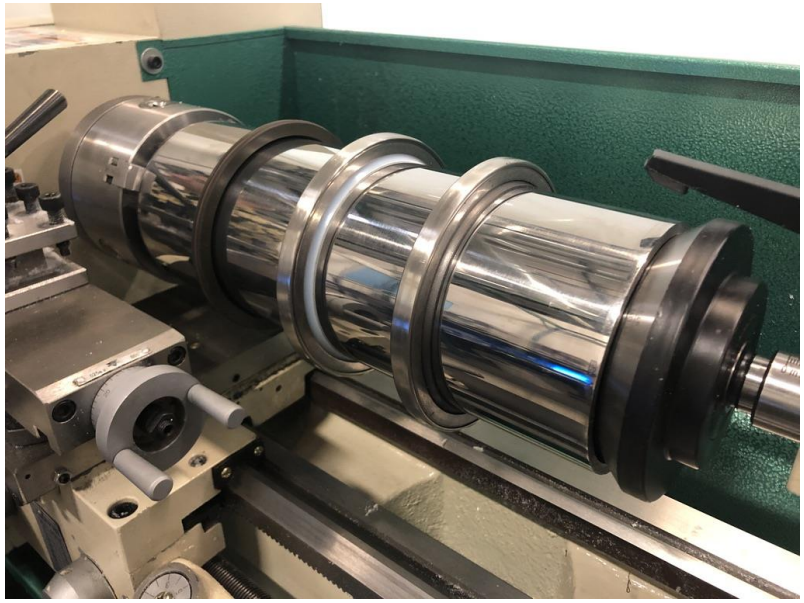
Relevance:

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**





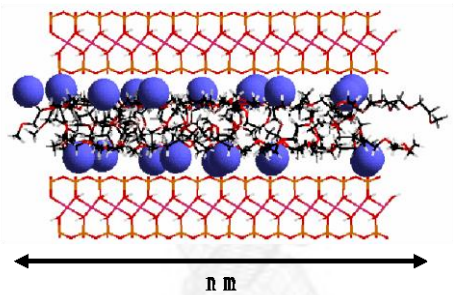
Reproduced 4.5" SS Shaft for Abrasion Testing of New Seal Materials to Mimic Beam Dump Water Seal

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

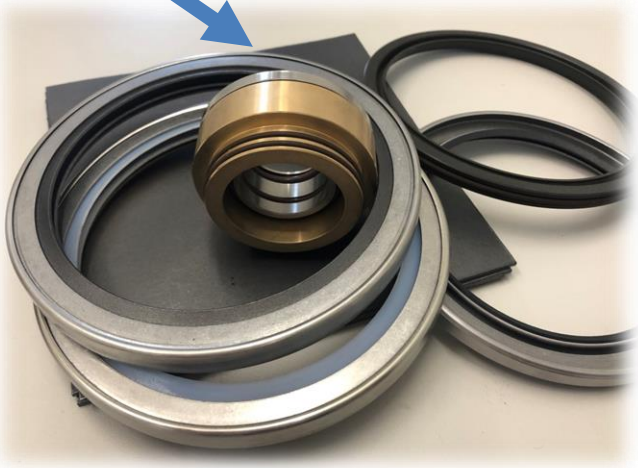


2. Design New Seals

- For new fittings
- Advanced materials
- Custom ID

<u>NEW SEAL</u>
Opposing lips
for
Water and Vacuum

Stationary seal
for
No rotation on drum

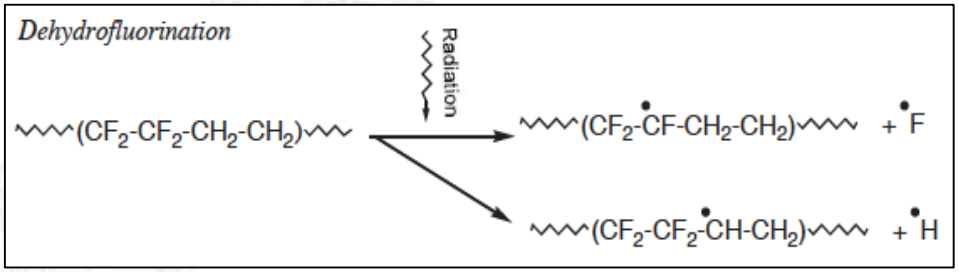


Current Seals
Material and Design Drawbacks

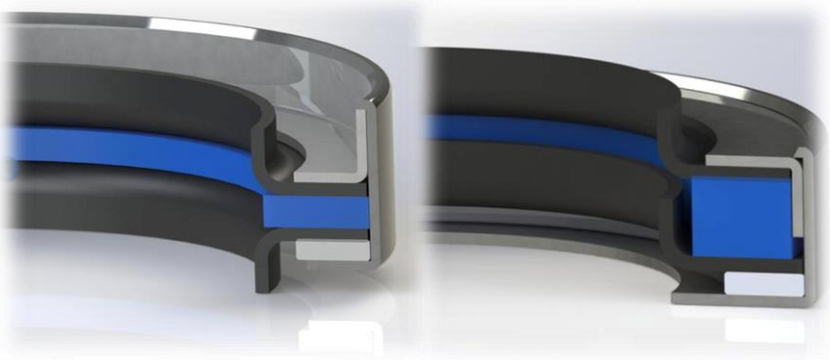
1. Material – Dehydrofluorination

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



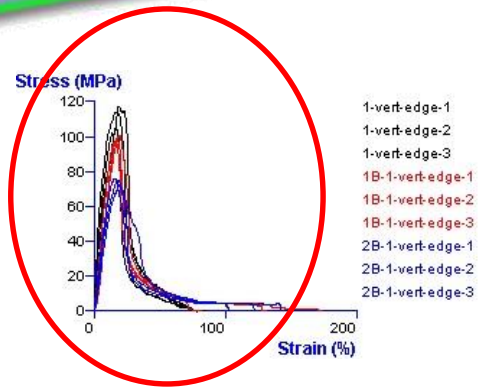
2. Seal – Need Opposing Lips

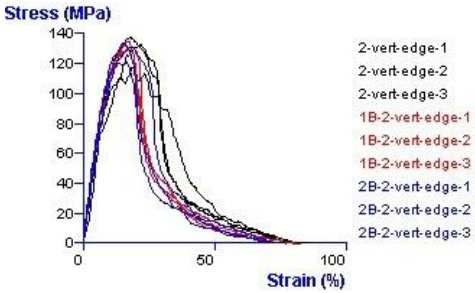


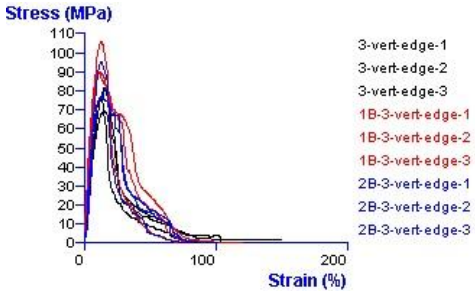
Or new design

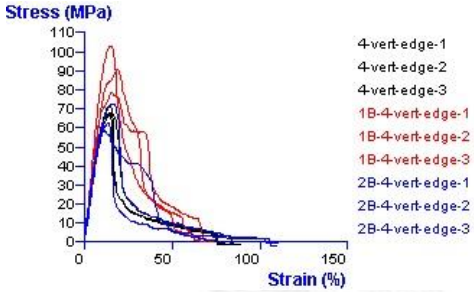
HF is typically carried off in vacuum,
may or may not be an issue for water in water cooled seals

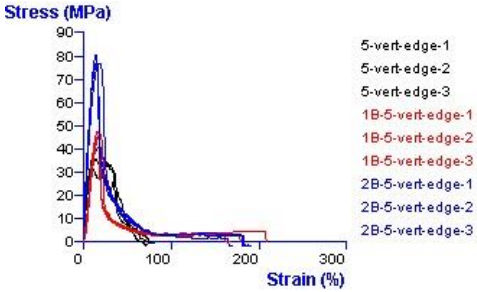
NanoSonic Vertical
Pre- and Post- Irradiation

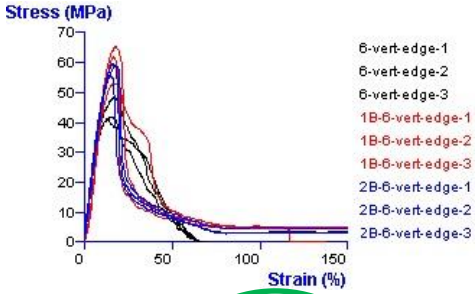


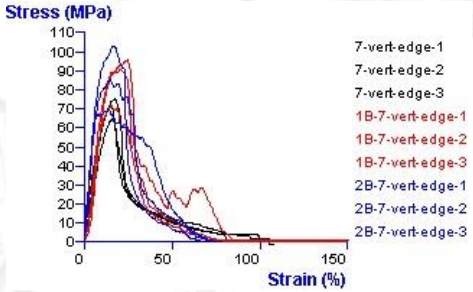


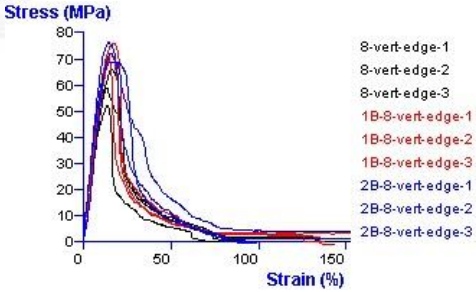


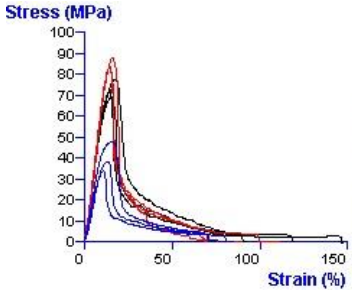


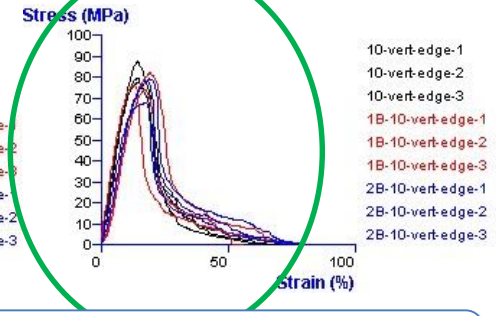








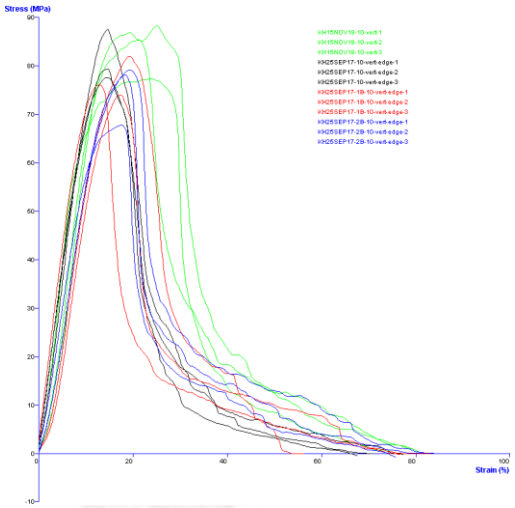


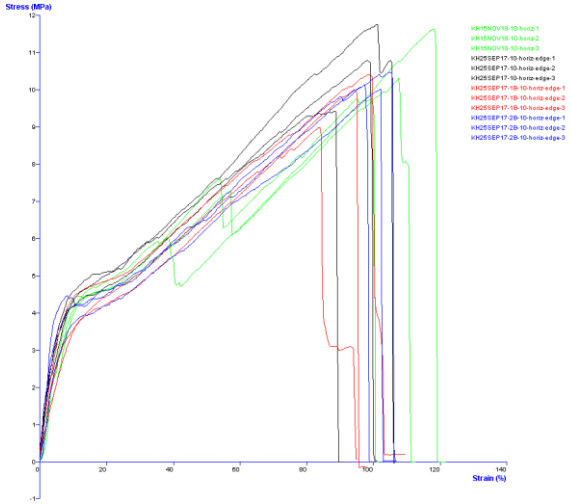


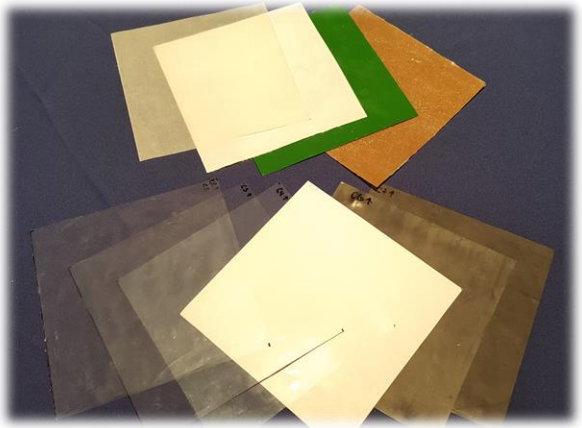
Note Tensile Strength Variations or Stability Post Fe and Proton Exposure

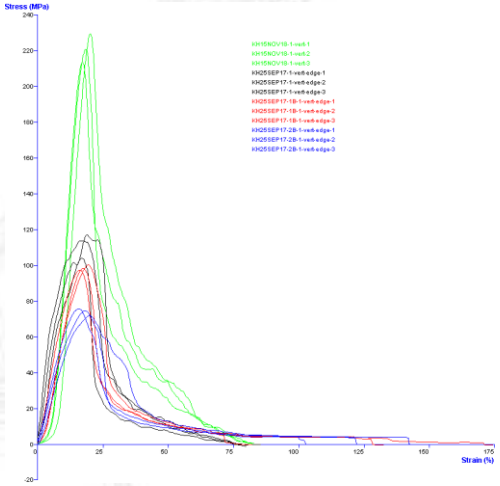
Accomplishments in Mechanical Properties Post Low Dose Exposure

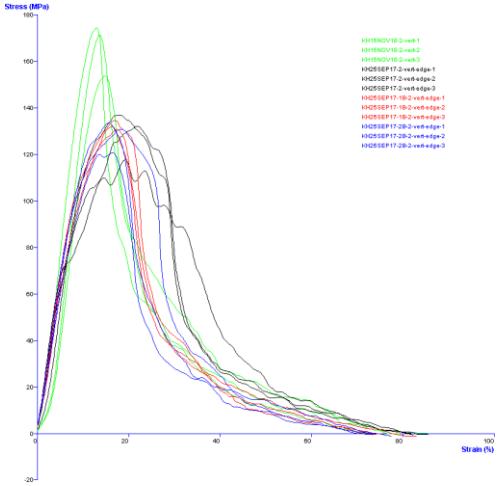
Electron, Fe, and Proton Irradiation – 27 Gy

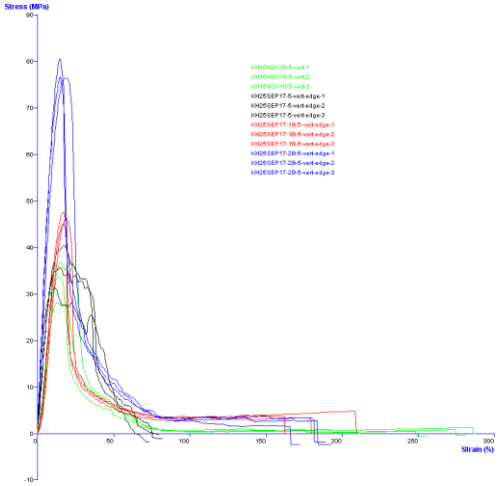












Tensile Strength Stability Post Fe, proton, and electron Exposure in both Directions

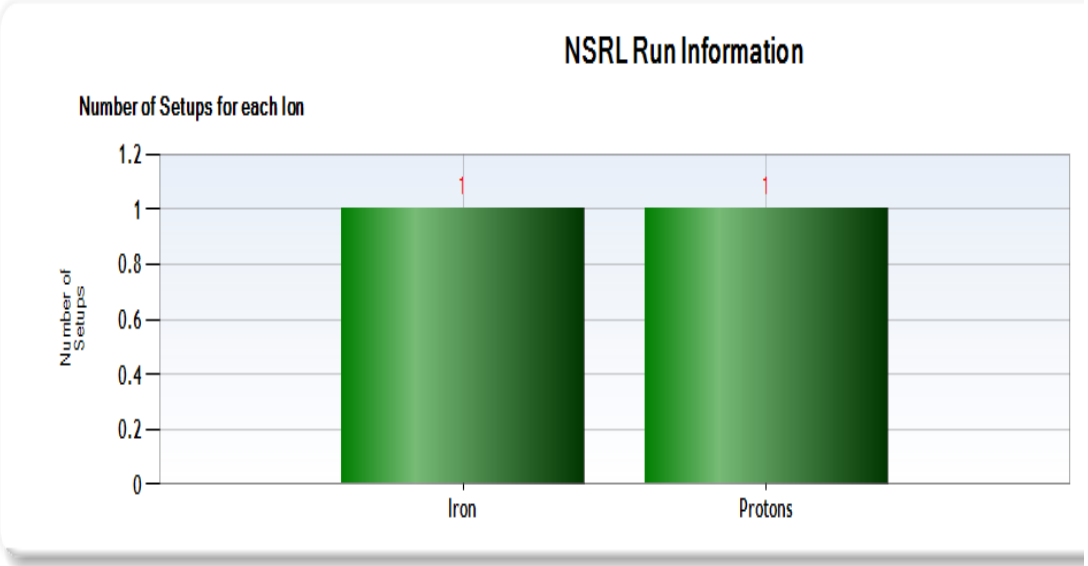
Accomplishments in Irradiation at BNL NSRL

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^2)	Particles Per Spill (particles/cm^2/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 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 304l is 30 vs. SS 304 of 70

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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

NanoSonic is Producing New Films for Use in Flood-Gard Seals

Acknowledgements

This material is based upon work supported by the Department of Energy, Office of Science, under Award No. DE-SC0017107

Dr. Jian Gao and Dr. Frederique Pellemoine MSU FRIB
Drs. Michelle Shinn and Elizabeth Bartosz, DOE

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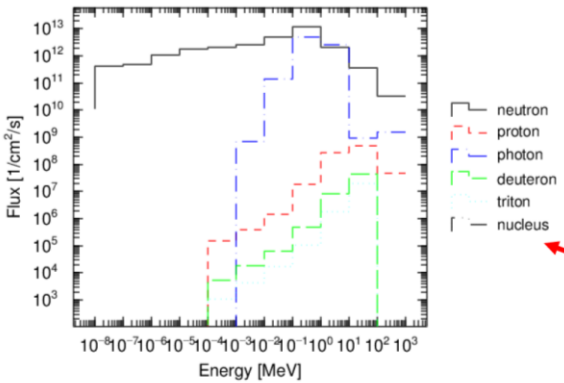
NanoSonic

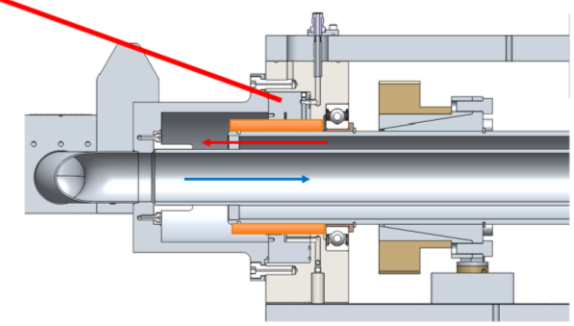
Accomplishments in High Dose Exposure

Plans for 600 MGy electron, proton, and Fe

- 48Ca at 400 kW, 261 MeV/u → 40Mg

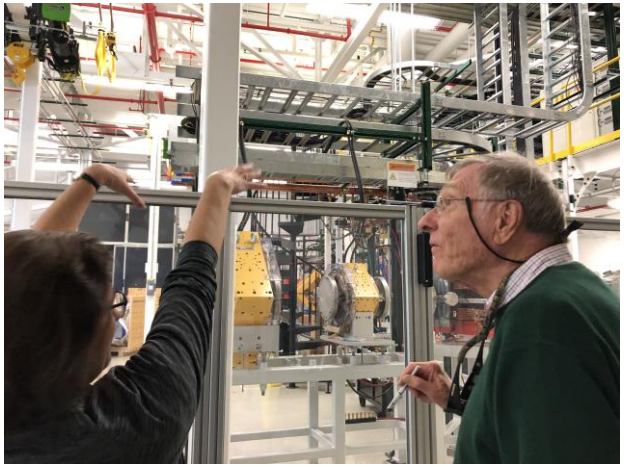


D. Georgobiani



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

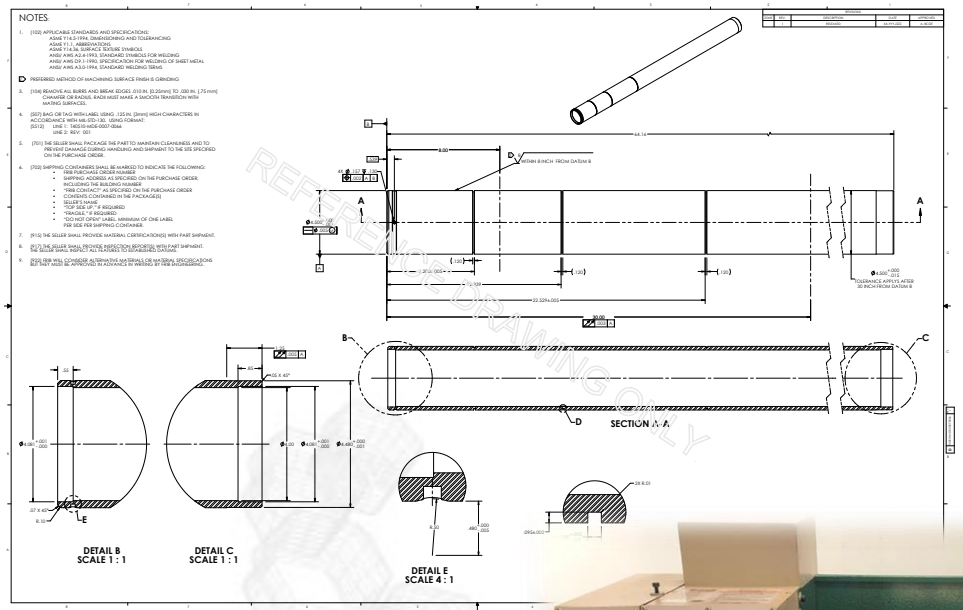
We will expose candidate seal materials to electron irradiation using high dose medical sterilization techniques at 50 MGy per pass for 100, 200, and 300 MGy doses at Steris – and BLIP



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

Accomplishments in Abrasion

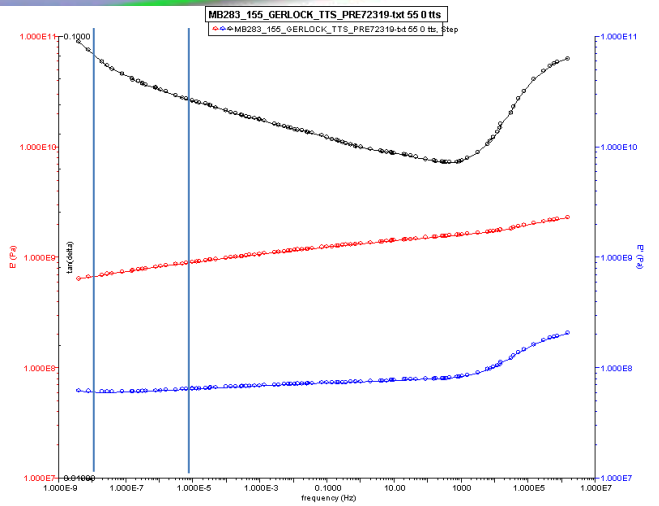
SS Shaft Developed for Seal Studies

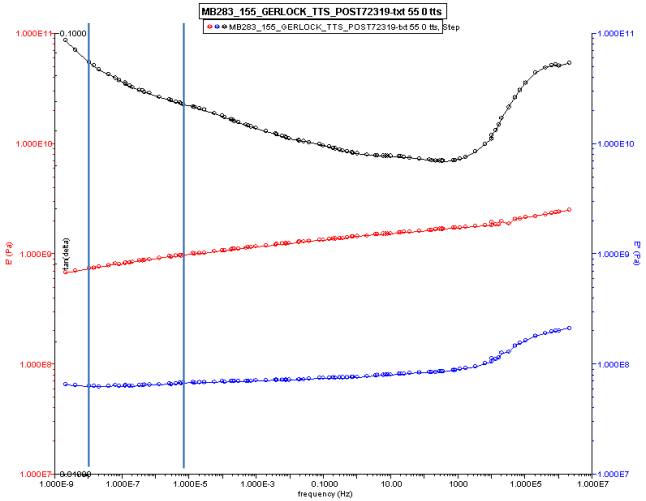


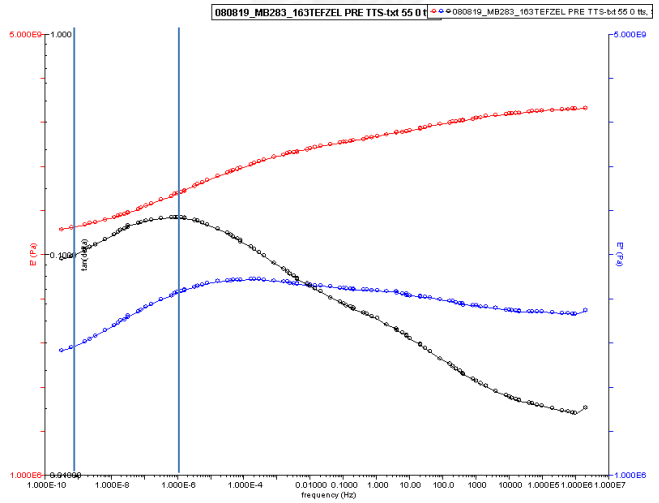
Developed SS Shaft per MSU Drawing for 600 rpm testing pre- and post- radiation exposure

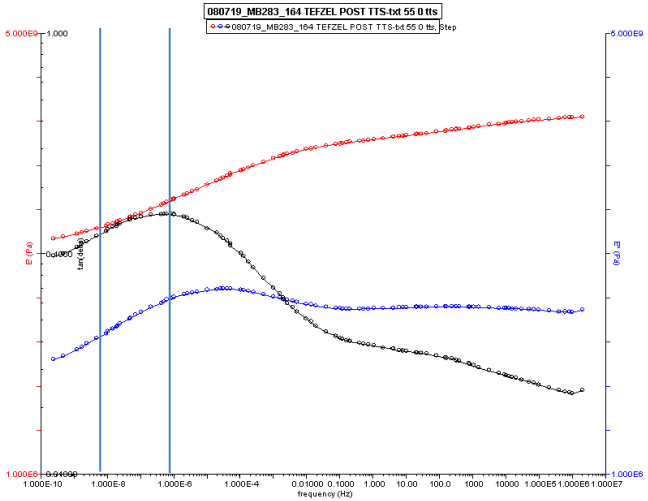
Accomplishments in Lifetime Assessment

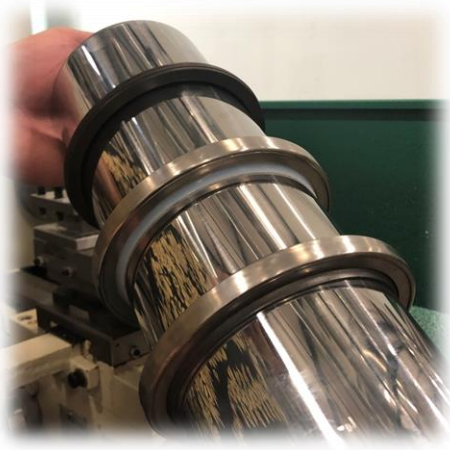
DMA TSS for 1-2 year predictive analysis









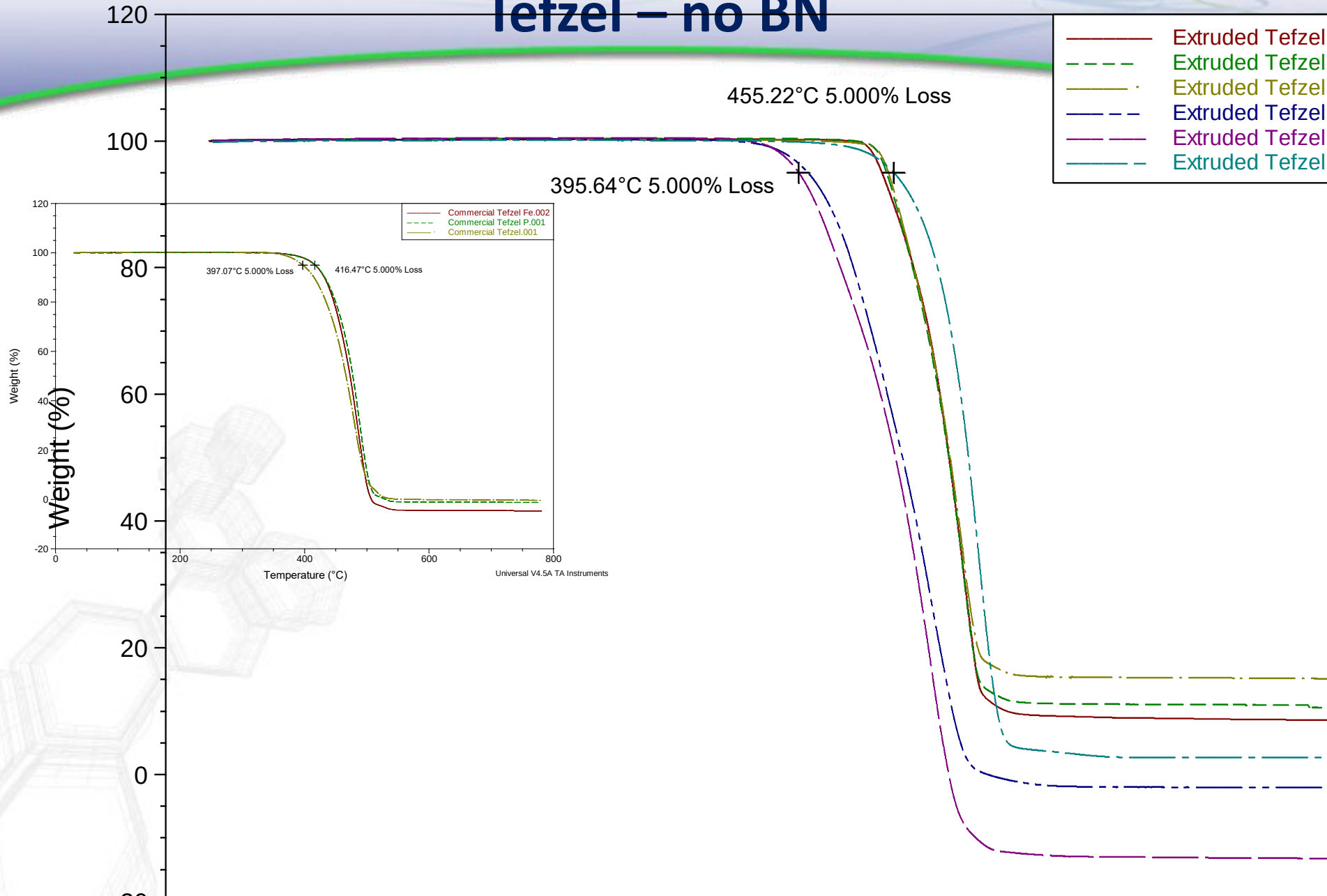


Temperature Step Frequency Sweep Runs Allow for Tan Delta and Loss Modulus Predictions at Instantaneous Events or out to > 2 Years

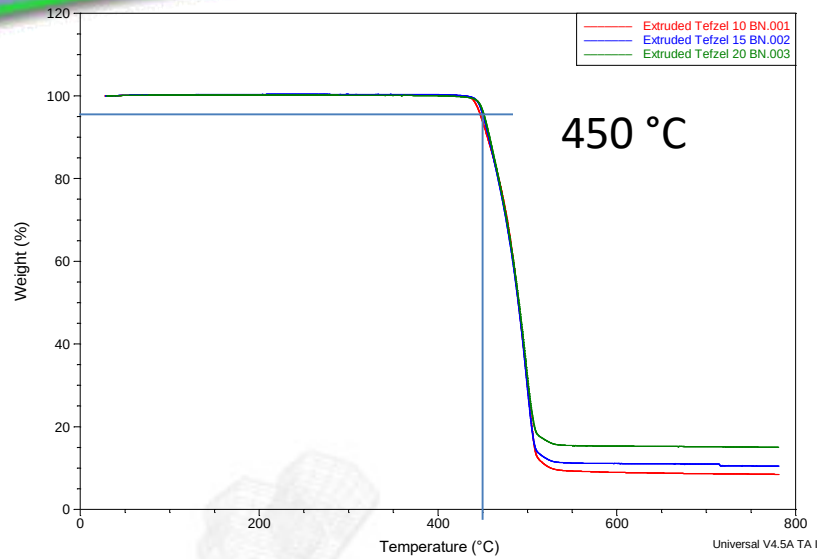
CODE

NSRL RUN		PROTON					IRON			
		ID	areal density (g/cm2)	mass density (g/cm3)	thickness (mm)		ID	areal density (g/cm2)	mass density (g/cm3)	thickness (mm)
NOV 2019	Garlock Teflon	26-p	0.35	2.15	1.25		26-Fe	0.35	2.17	1.63
	Garlock Gylon	27a-p	0.32	2.11	1.21		27a-Fe	0.32	2.10	1.54
	Garlock Gylon	27b-p	0.32	2.10	1.21		27b-Fe	0.32	2.09	1.55
	Tefzel 0.093"	28-p	0.44	1.70	1.07		28-Fe	0.44	1.70	2.60
	PE L run	29-p	0.14	0.90	0.52		29-Fe	0.14	0.91	1.57
	PE - 5% BN L run	30-p	0.19	0.97	0.58		30-Fe	0.19	0.98	1.96
	PE - 20% BN L run	31-p	0.14	0.95	0.54		31-Fe	0.14	0.99	1.44
	PE - 50% BN L run	32-p	0.11	1.21	0.66		32-Fe	0.11	1.27	0.89
	Es L run	33-p	0.23	1.10	0.66		33-Fe	0.22	1.10	2.03
	Es - 10 % BN L run	34-p	0.22	1.13	0.68		34-Fe	0.23	1.15	1.99
	Es - 50 % BN L run	35-p	0.24	1.32	0.78		35-Fe	0.24	1.37	1.75
MAR 2020	ET N run	ET	0.17	1.56	1.07		ET	0.17	1.55	1.07
	ET - 10% BN	ET-10	0.12	1.69	0.69		ET-10	0.12	1.72	0.72
	ET - 15% BN	ET-15	0.12	1.62	0.85		ET-15	0.13	1.57	0.73
	ET - 20% BN	ET-20	0.15	1.73	0.76		ET-20	0.14	1.79	0.85

Thermal Testing
Tefzel – no BN

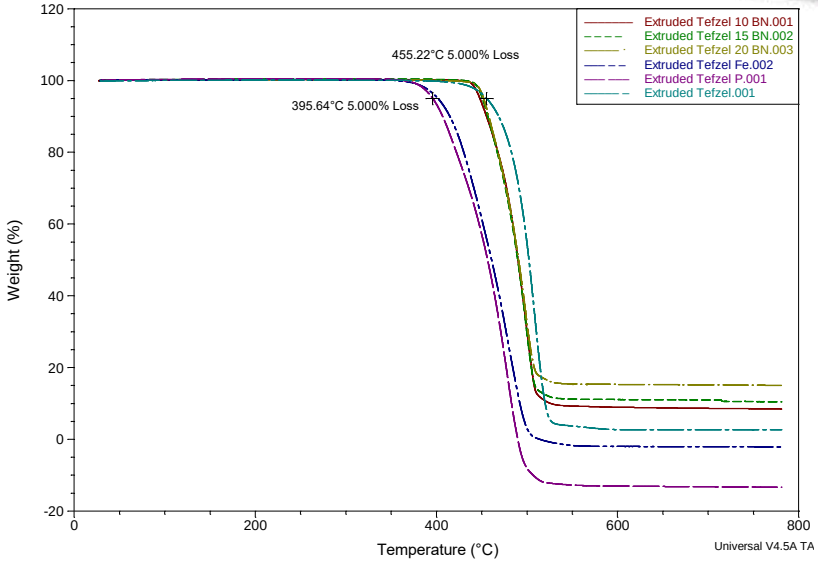


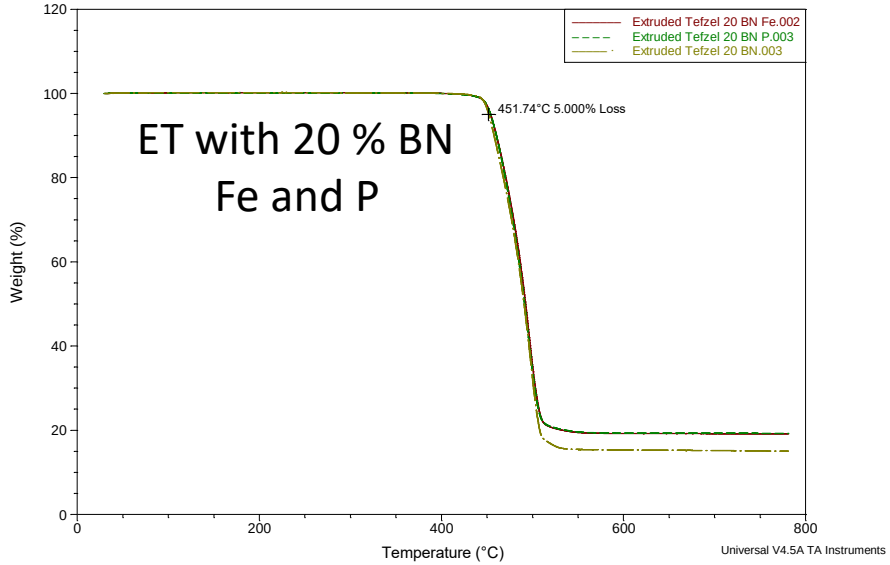
Thermal Testing
Tefzel



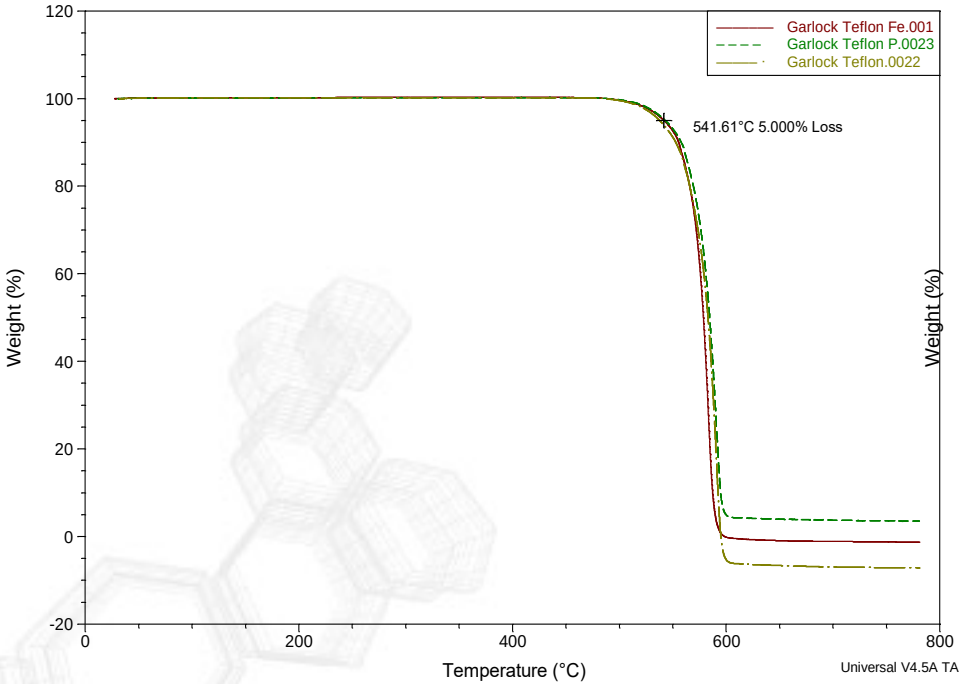
Show photo of tefzel BN films

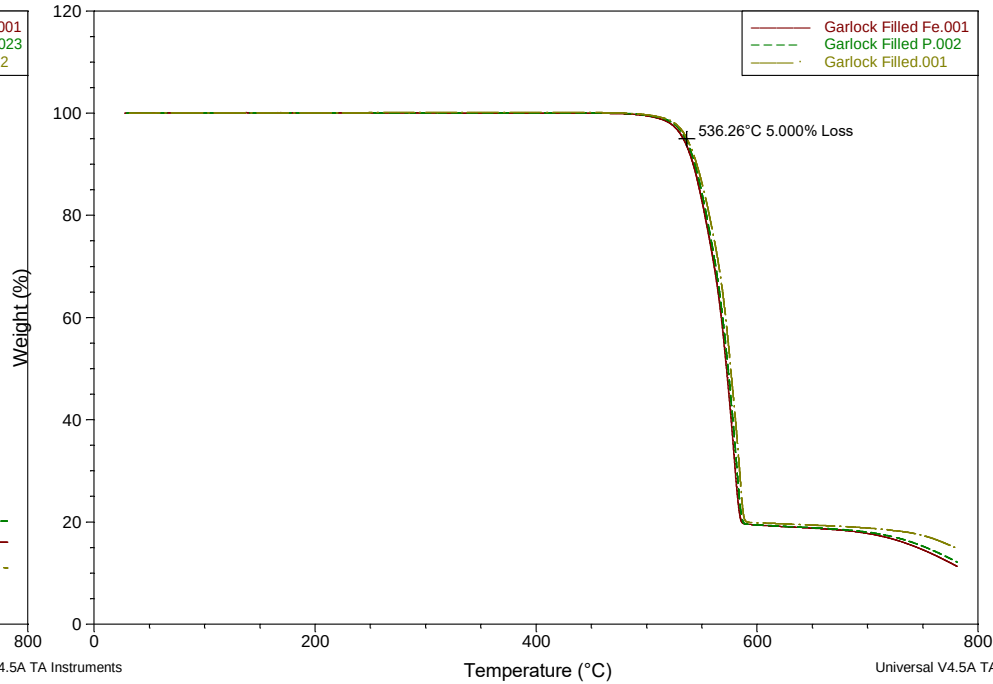
Thermal Testing
Tefzel



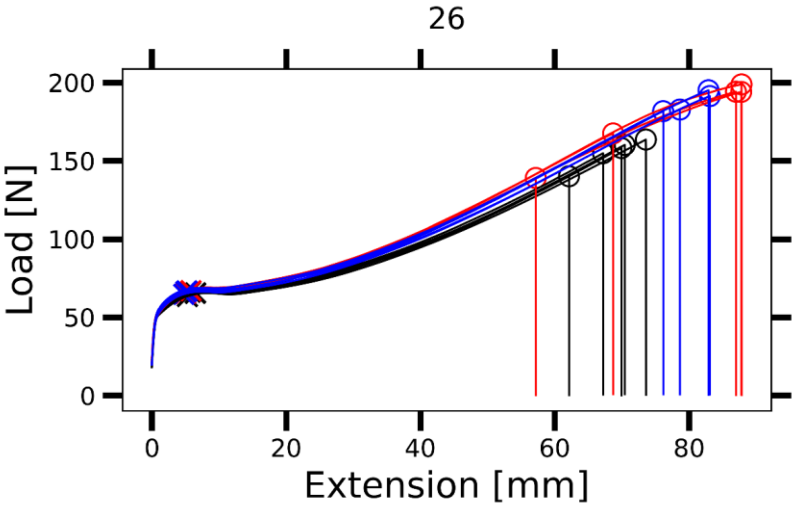


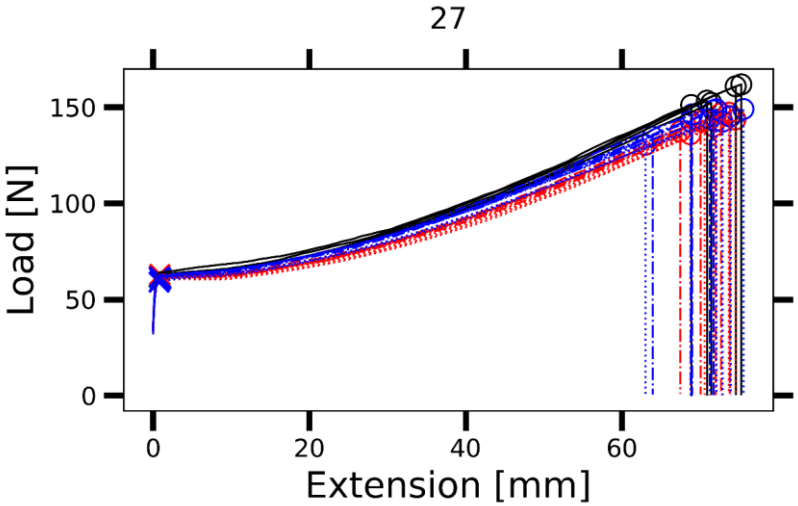
Thermal Testing
Teflon

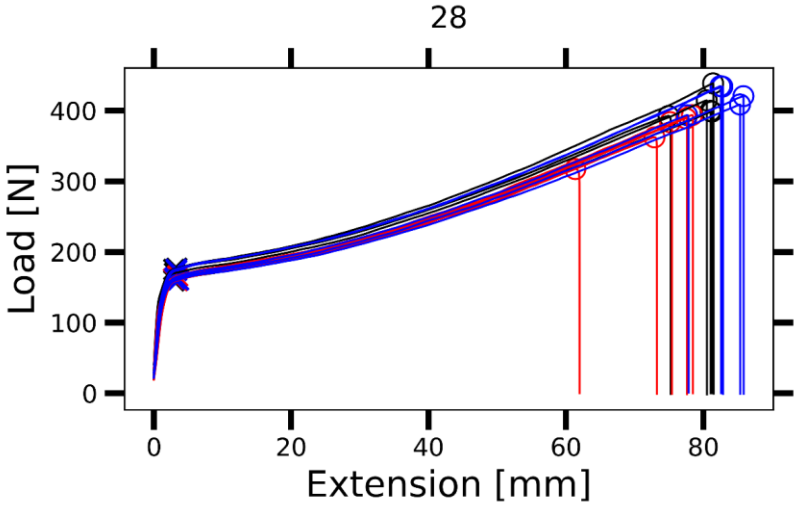




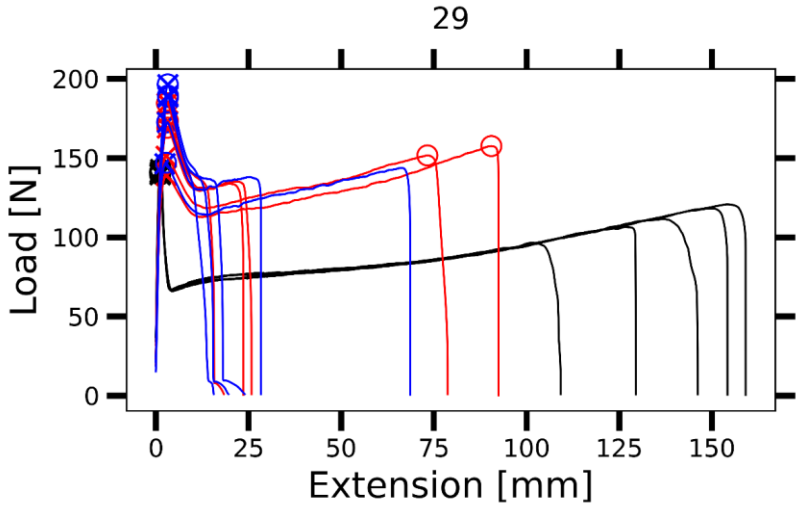
Mechanical Testing
26, 27, 28 - Room Temp

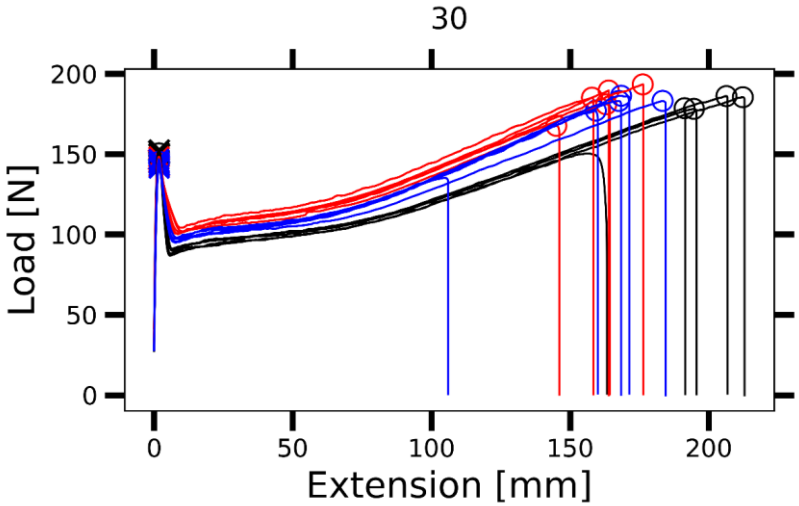


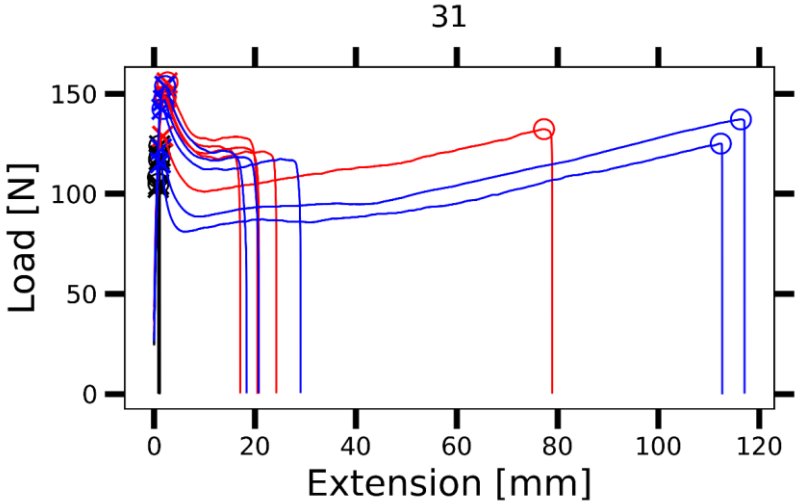


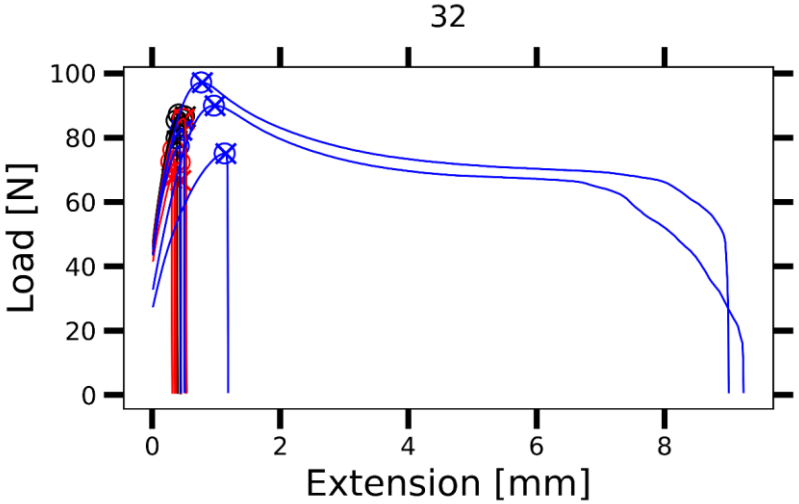


Mechanical Testing
29, 30, 31, 32 - Room Temp

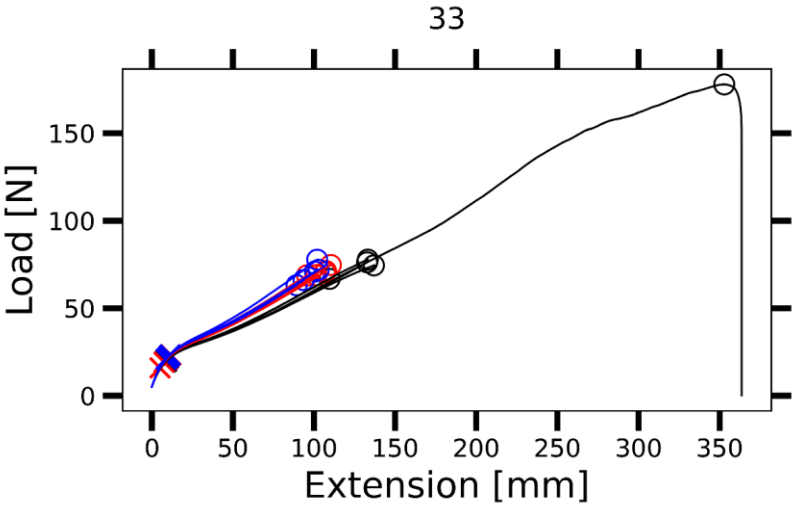


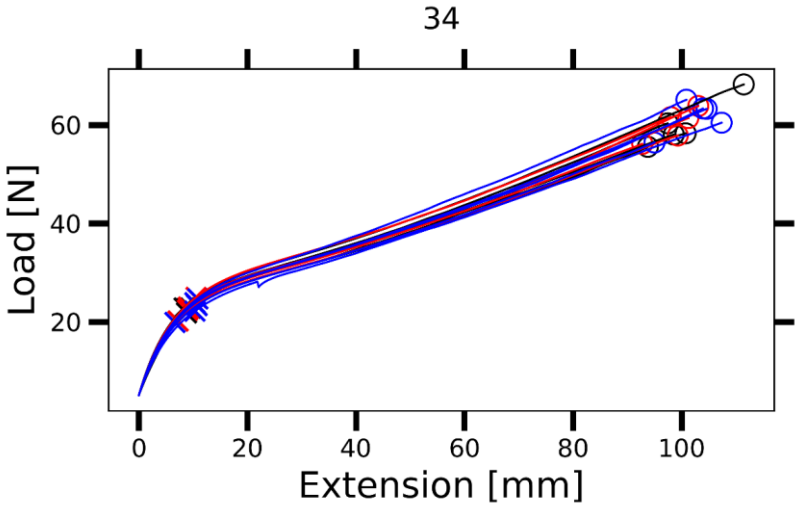


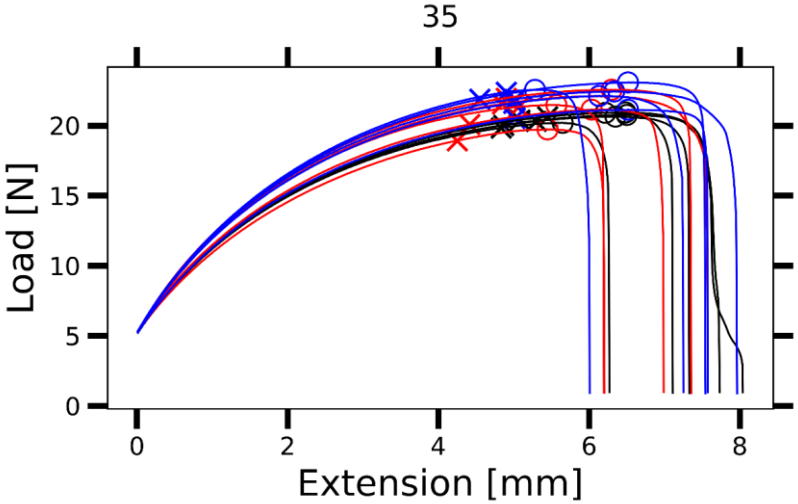




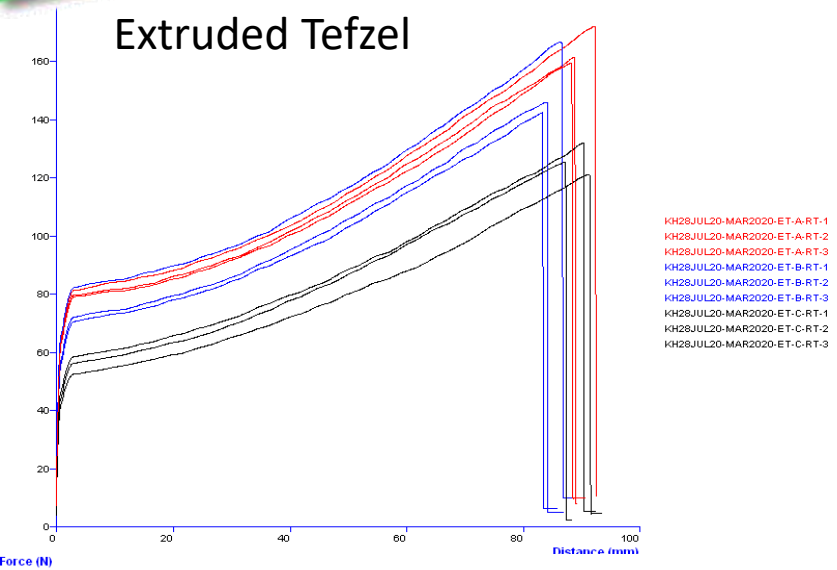
Mechanical Testing
33, 34, 35 - Room Temp

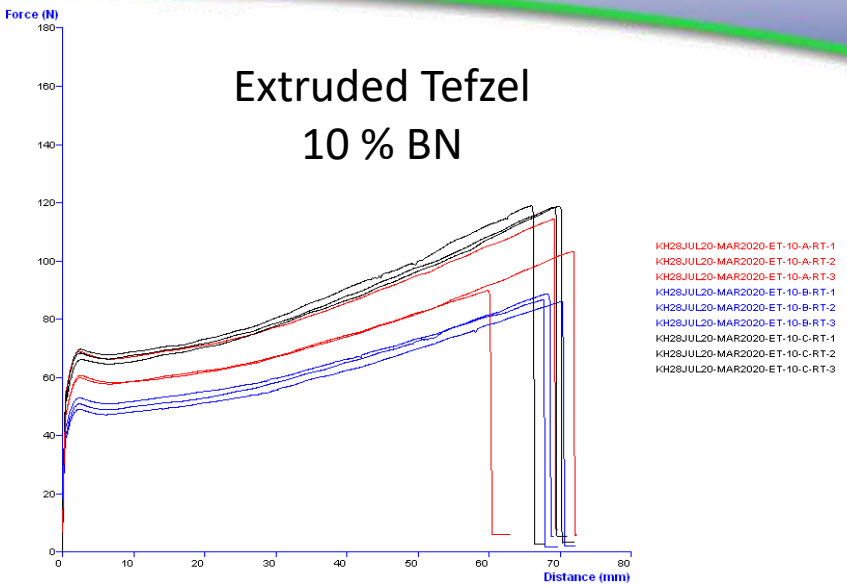


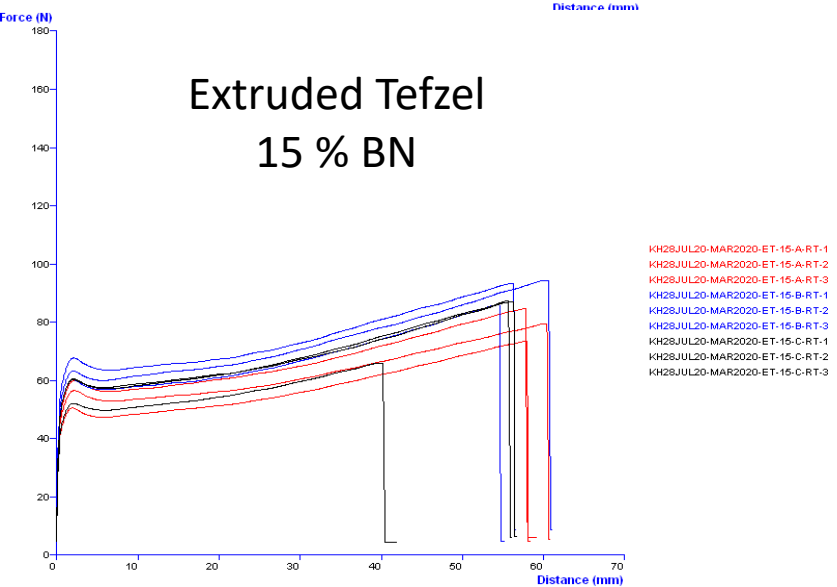


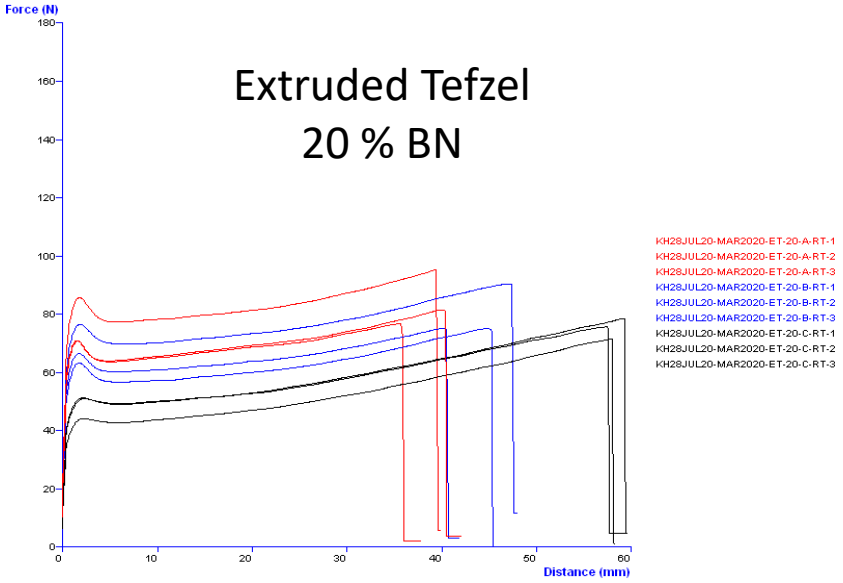


Mechanical Testing:
Extruded Tefzel (ET) with BN at Room Temp

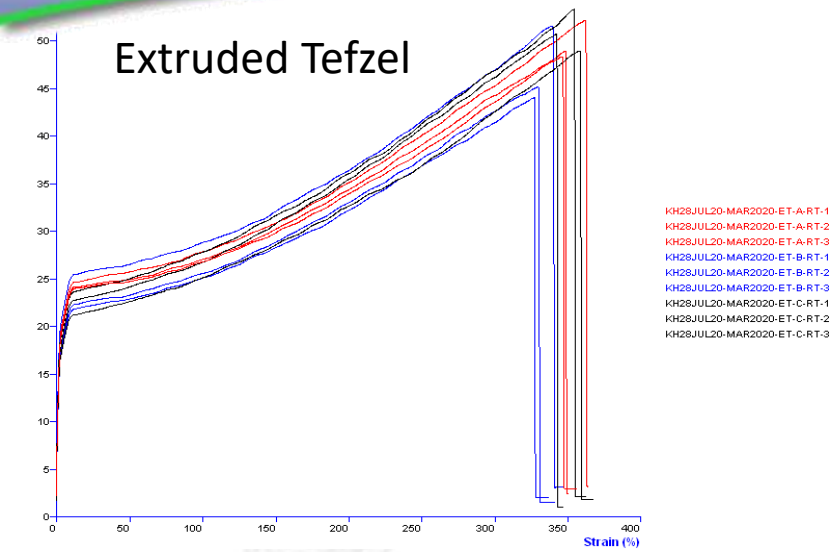


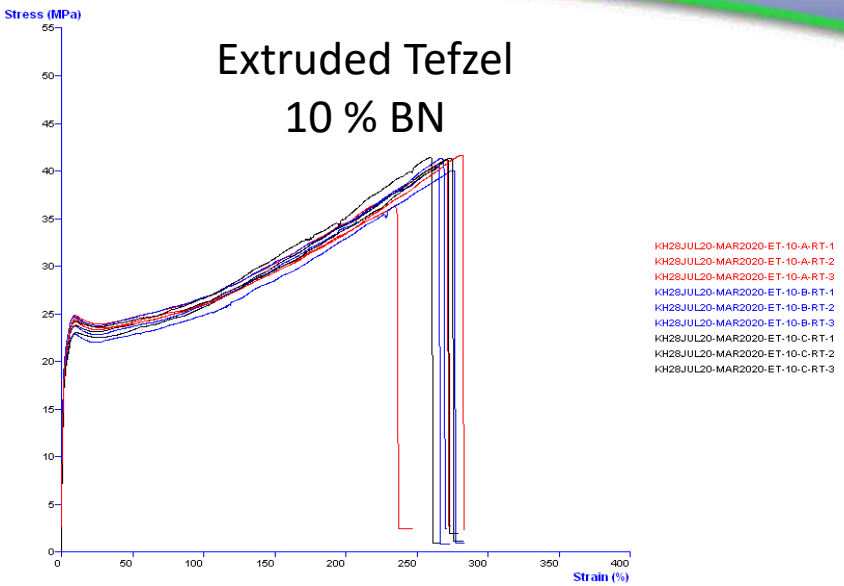


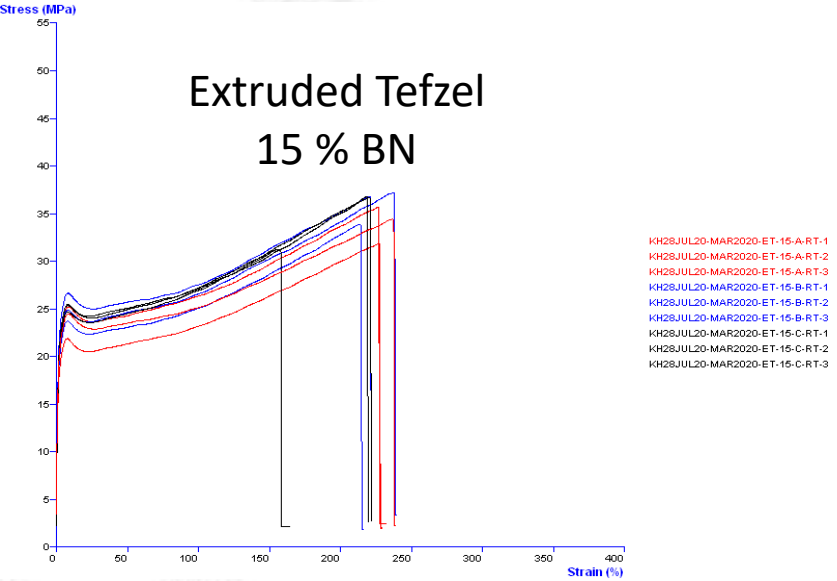


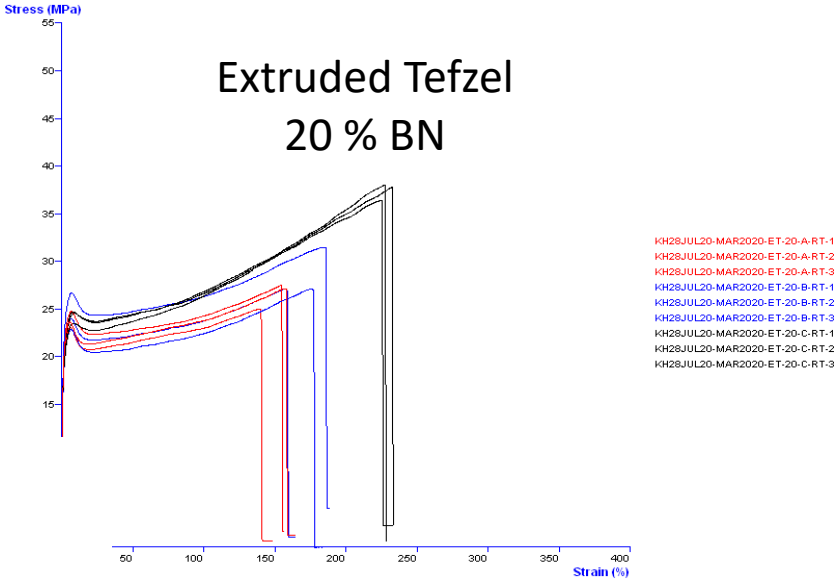


Mechanical Testing:
Extruded Tefzel (ET) with BN at Room Temp

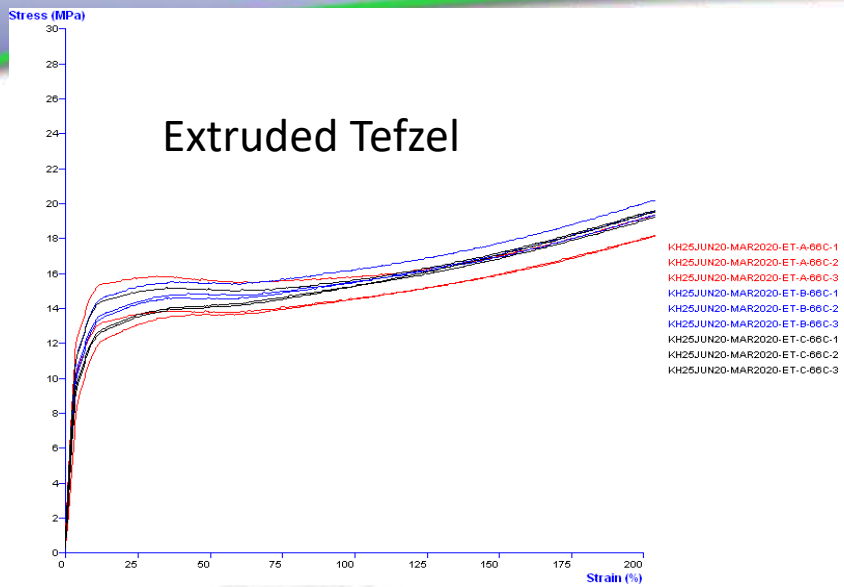


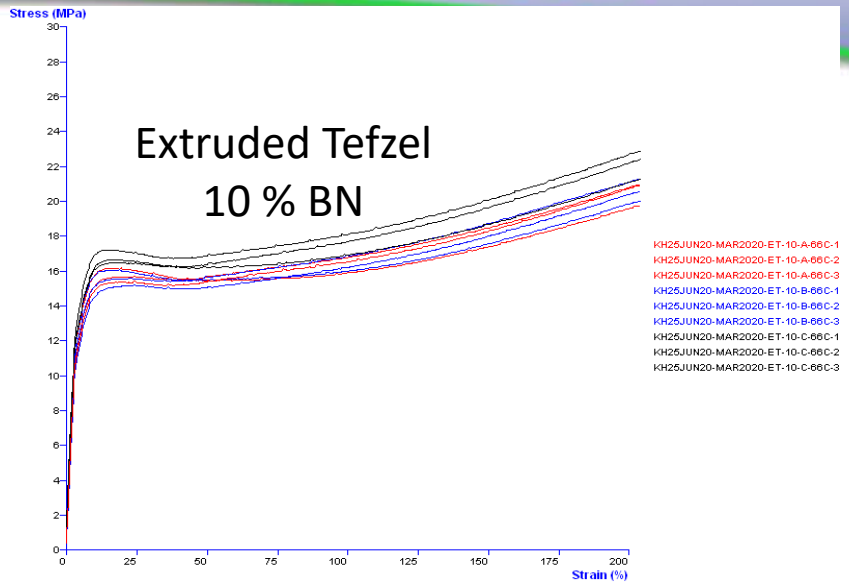


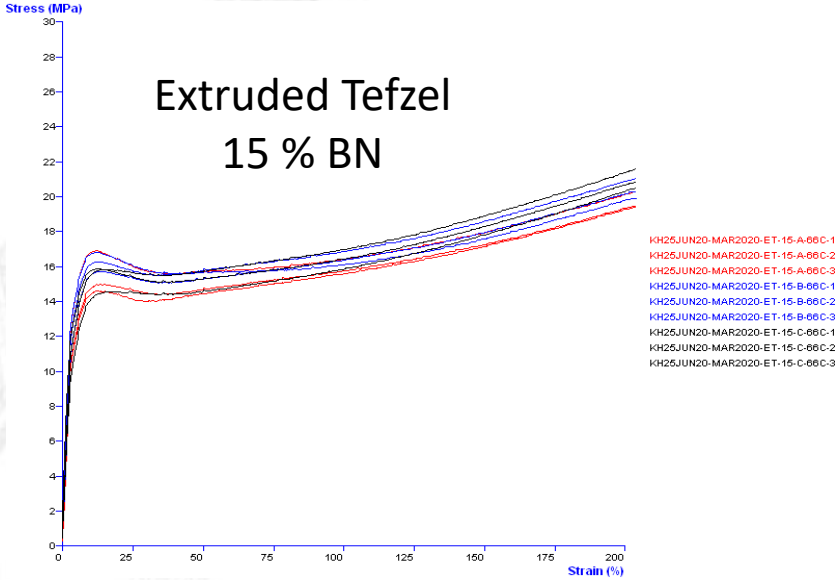


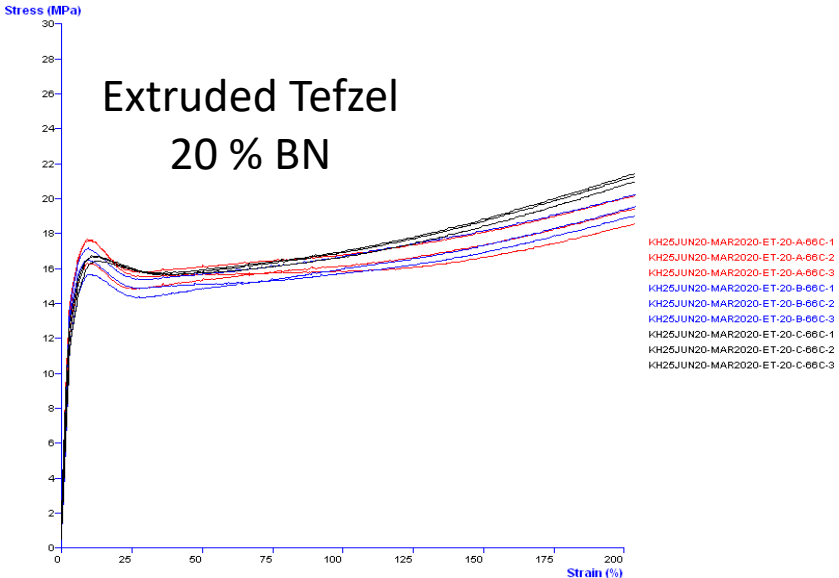


Mechanical Testing:
Extruded Tefzel (ET) with BN at 66 °C

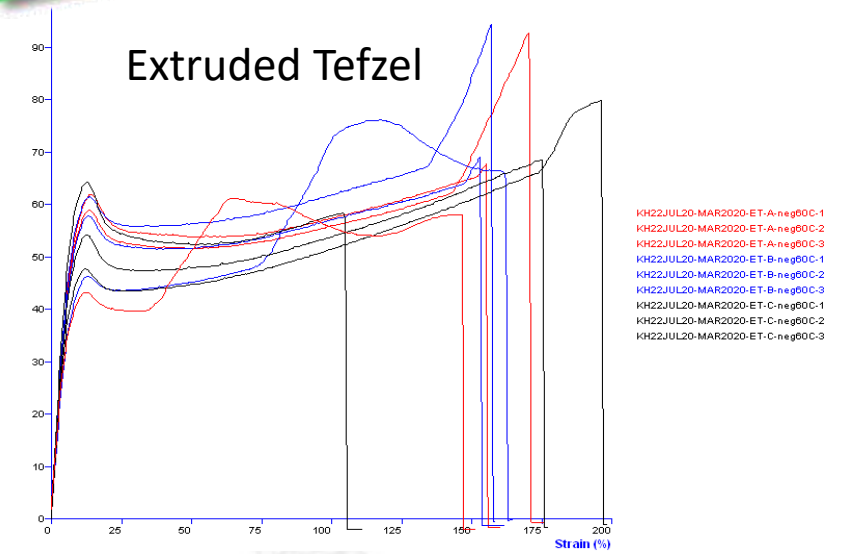


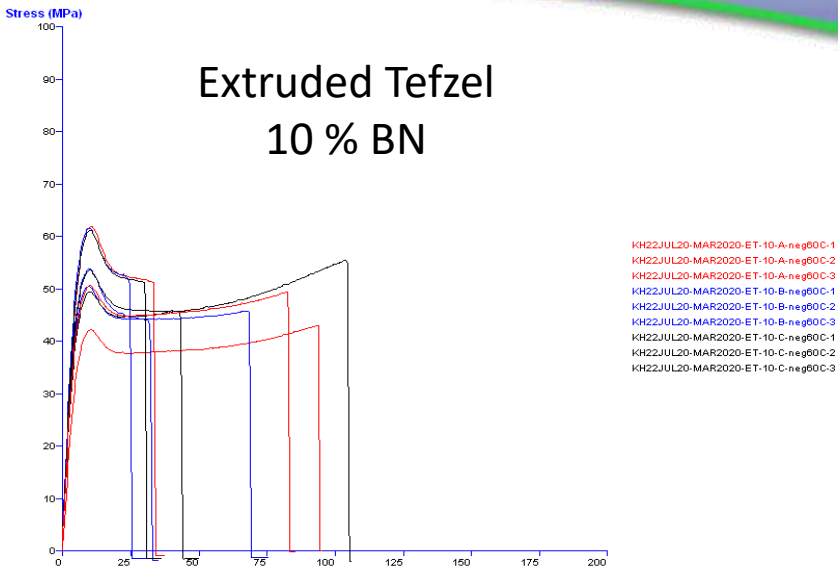


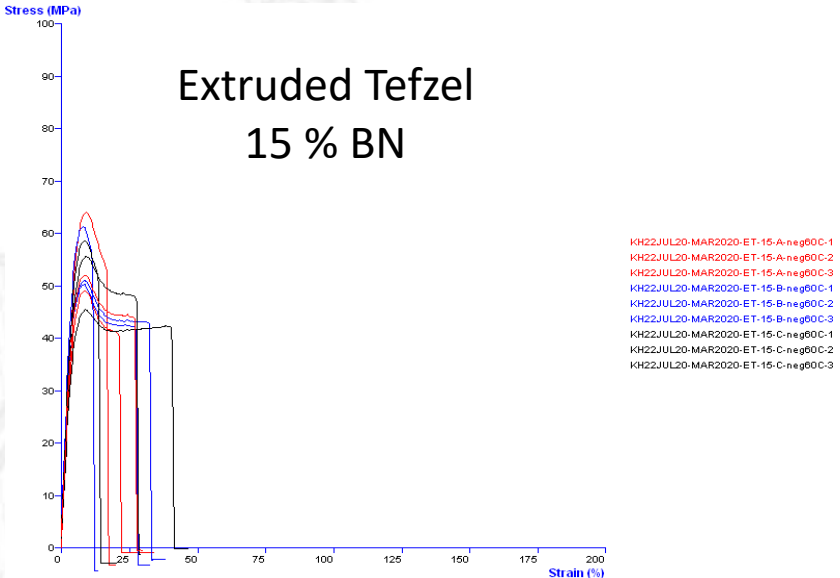


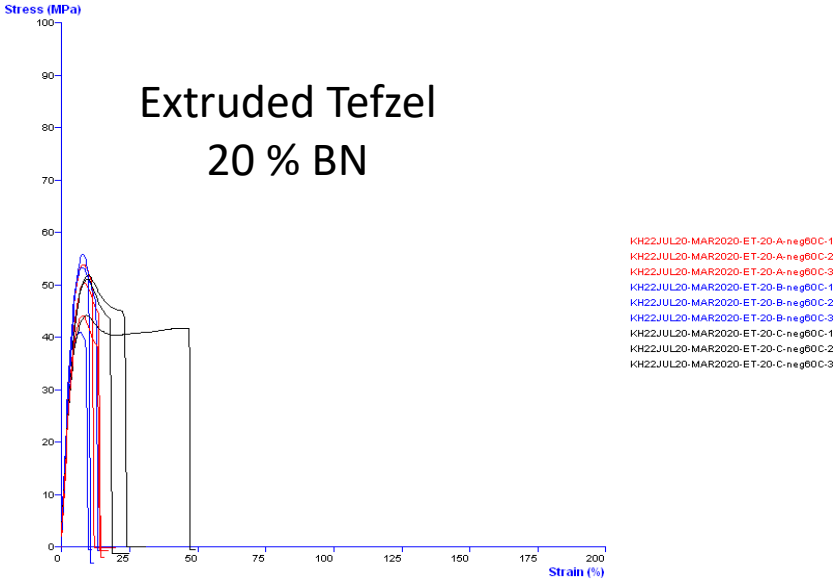


Mechanical Testing:
Extruded Tefzel (ET) with BN at -60 °C

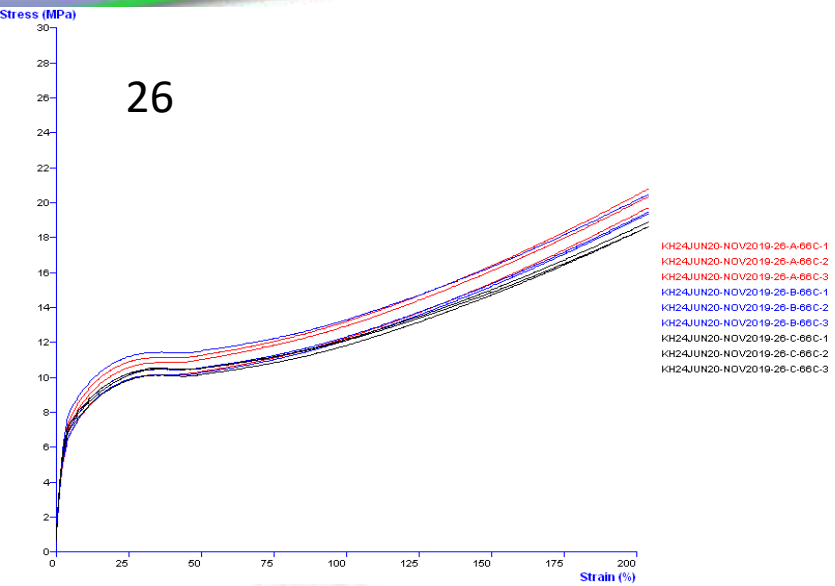


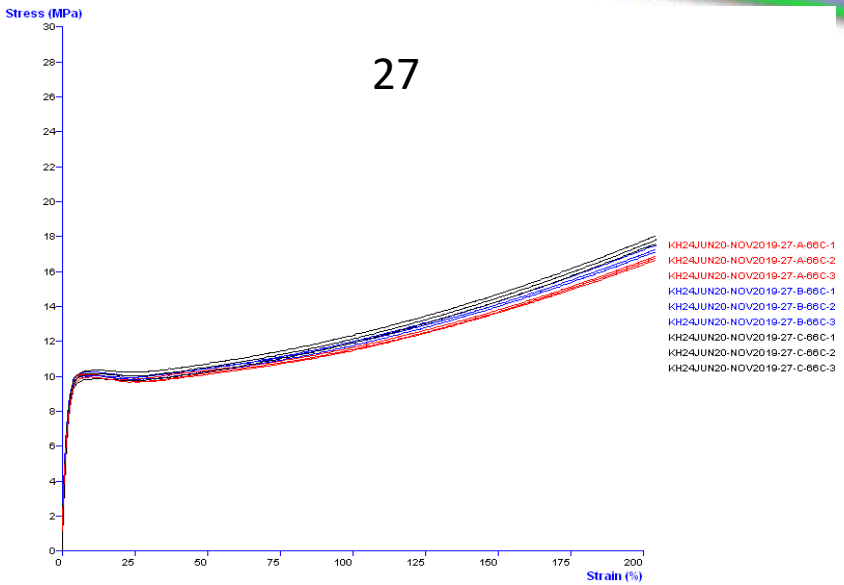


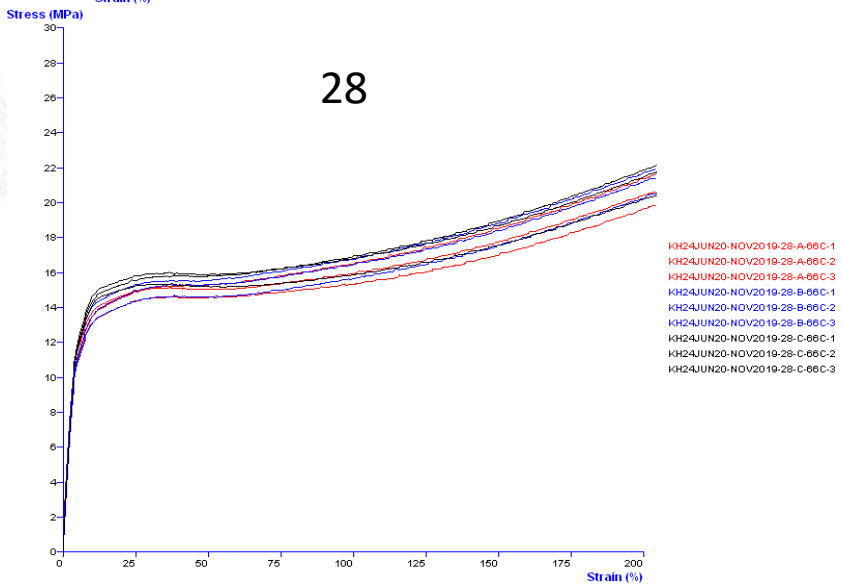




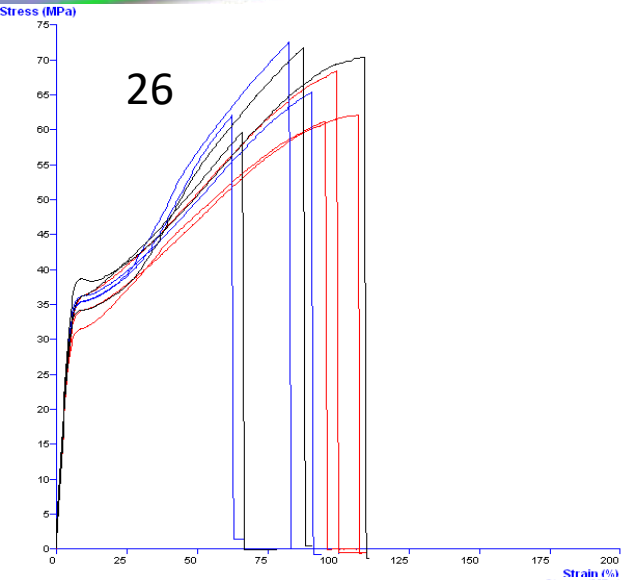
Mechanical Testing for 26, 27, 28: November 2019 run at 66 °C

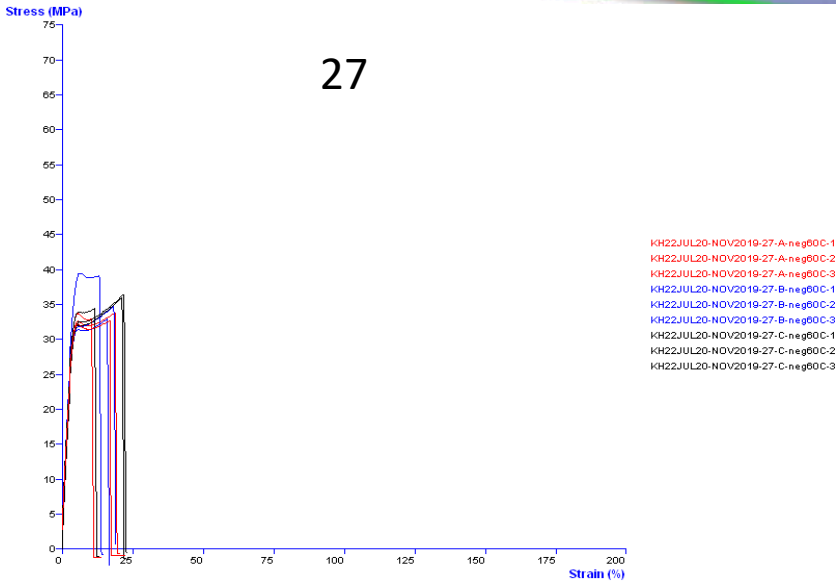


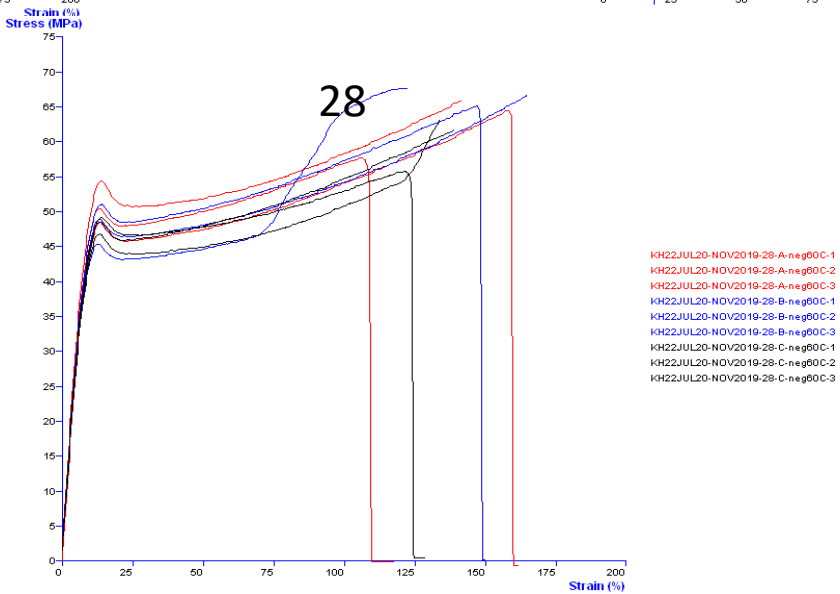




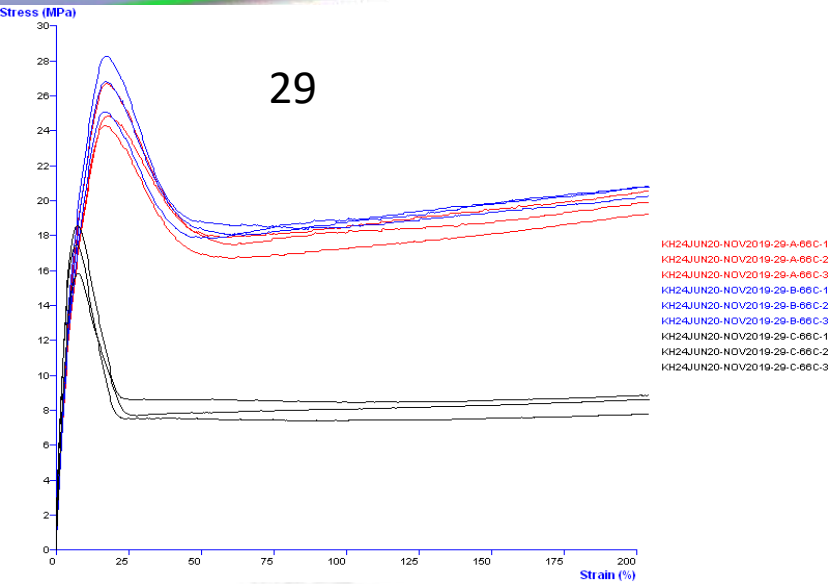
Mechanical Testing for 26, 27, 28: November 2019 run at -60 °C

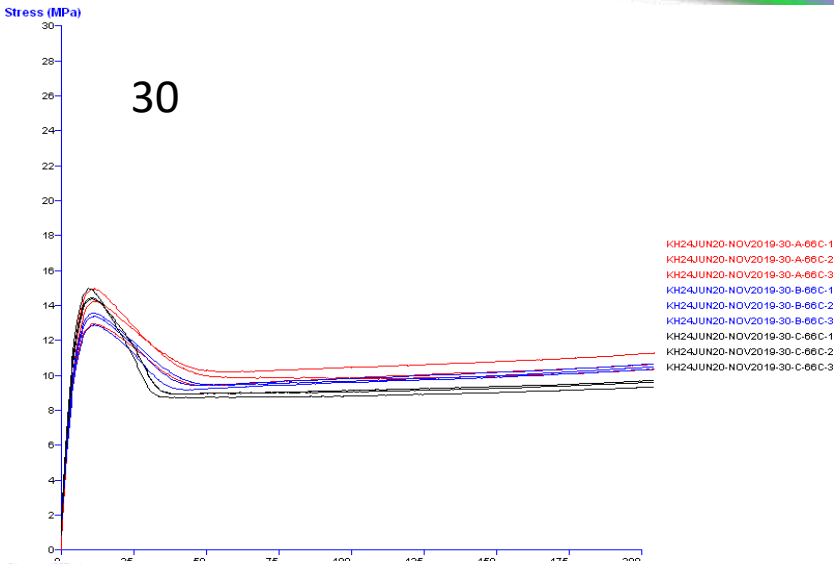


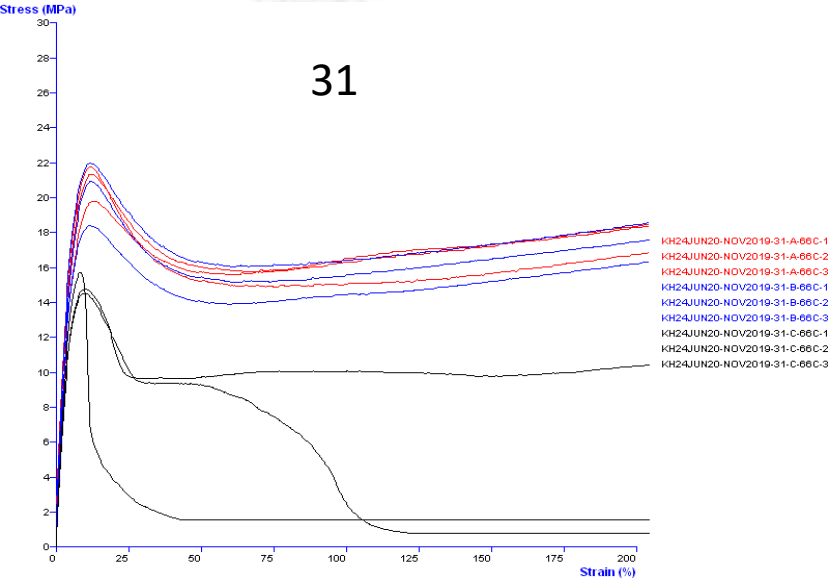


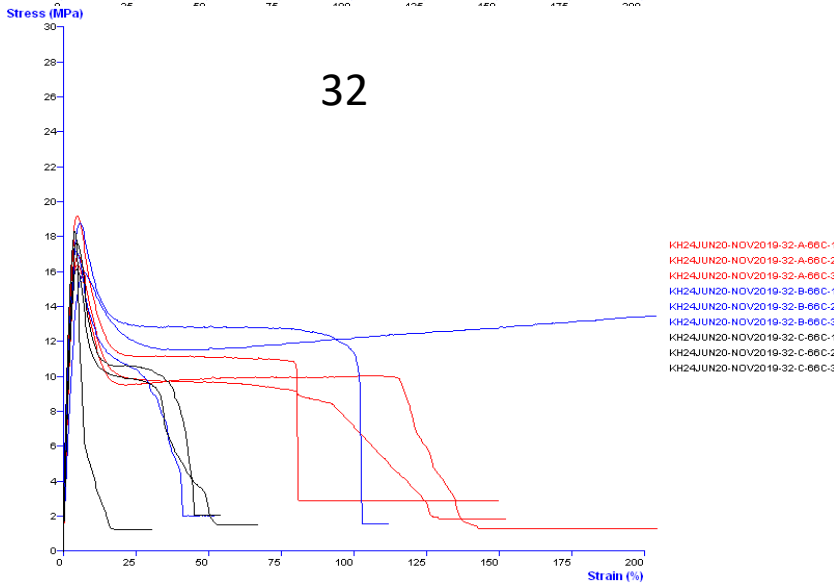


Mechanical Testing for 29, 30, 31, 32: November 2019 run at 66 °C

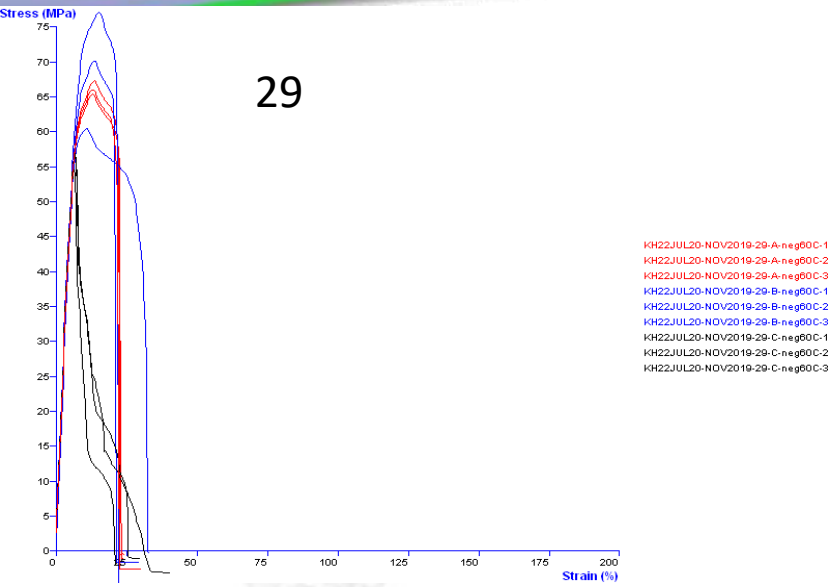


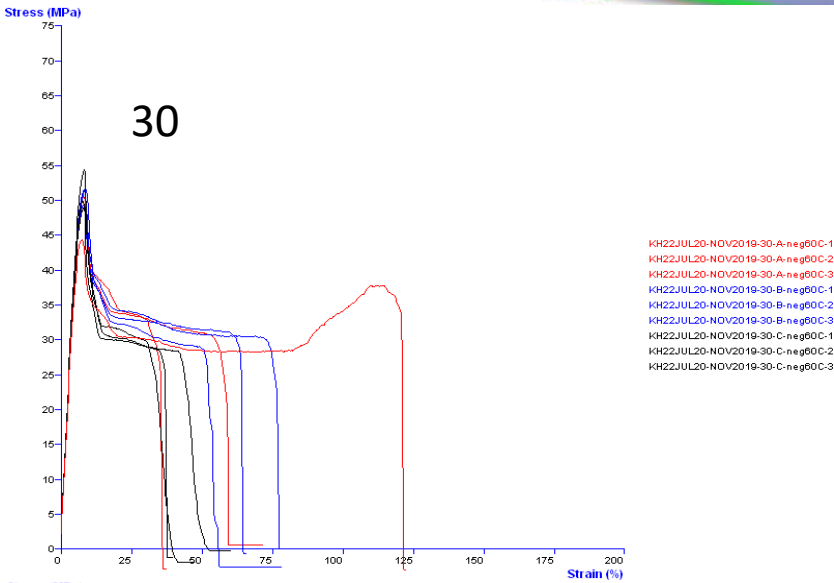


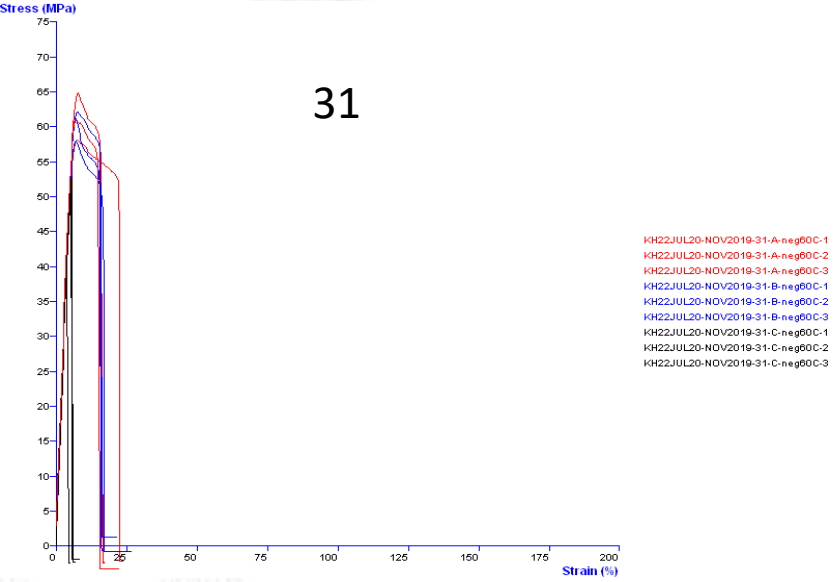


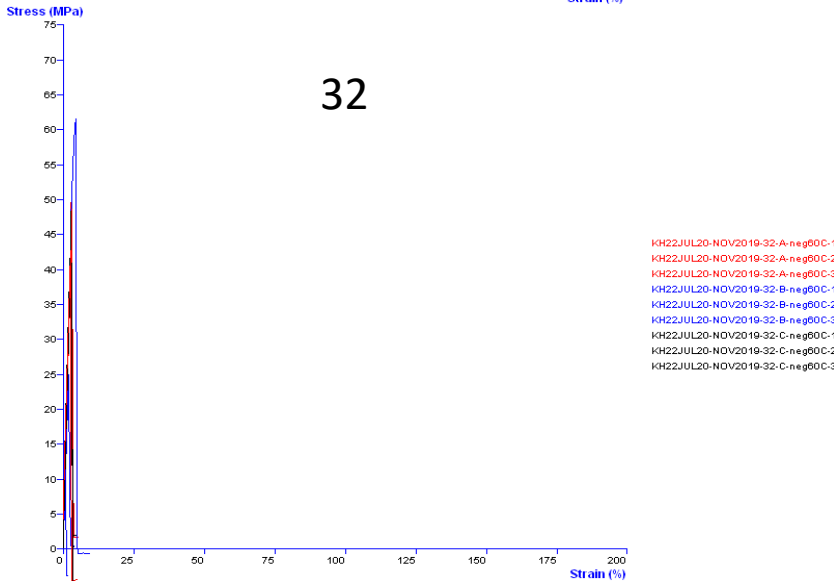


Mechanical Testing for 29, 30, 31, 32: November 2019 run at -60 °C



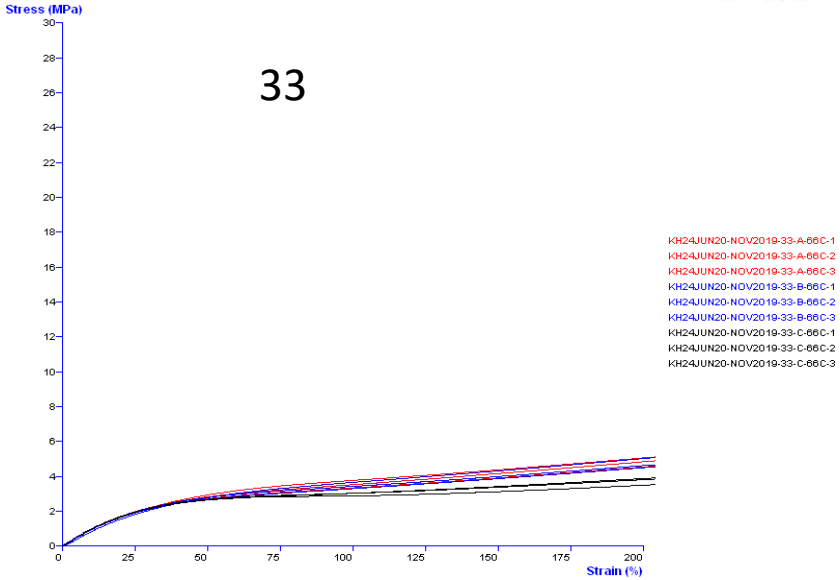




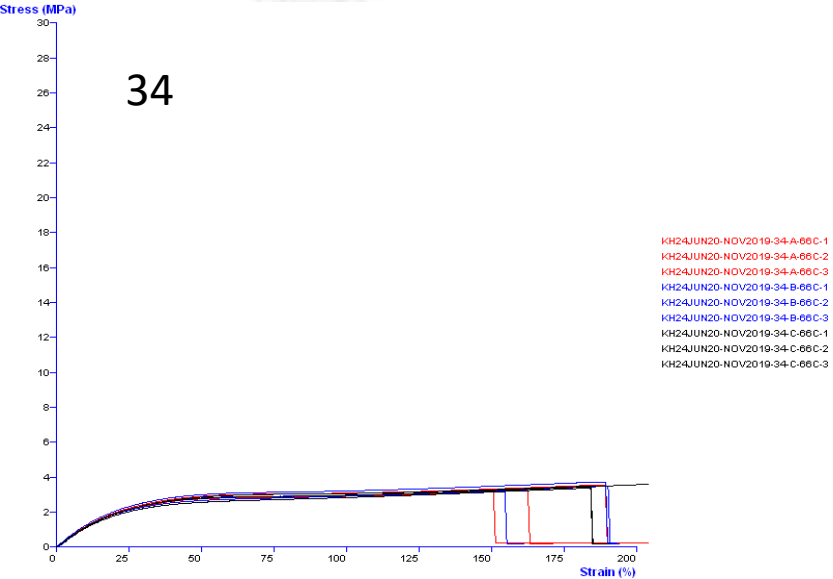


Mechanical Testing for 33, 34, 35 November 2019 run at 66 °C

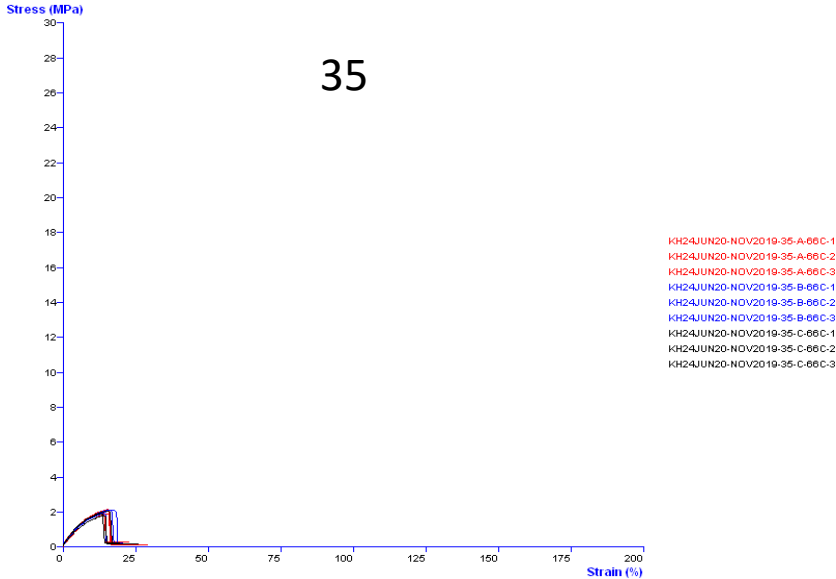
33



34

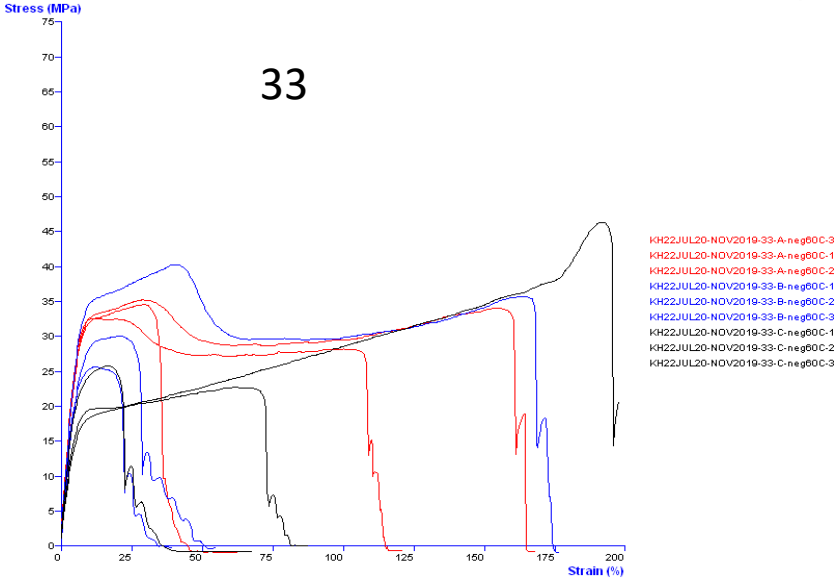


35

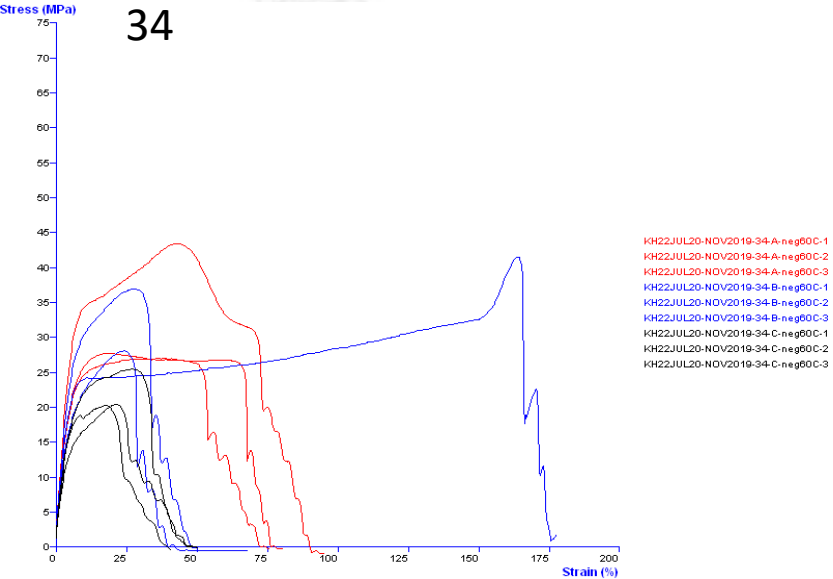


Mechanical Testing for 33, 34, 35 November 2019 run at -60 °C

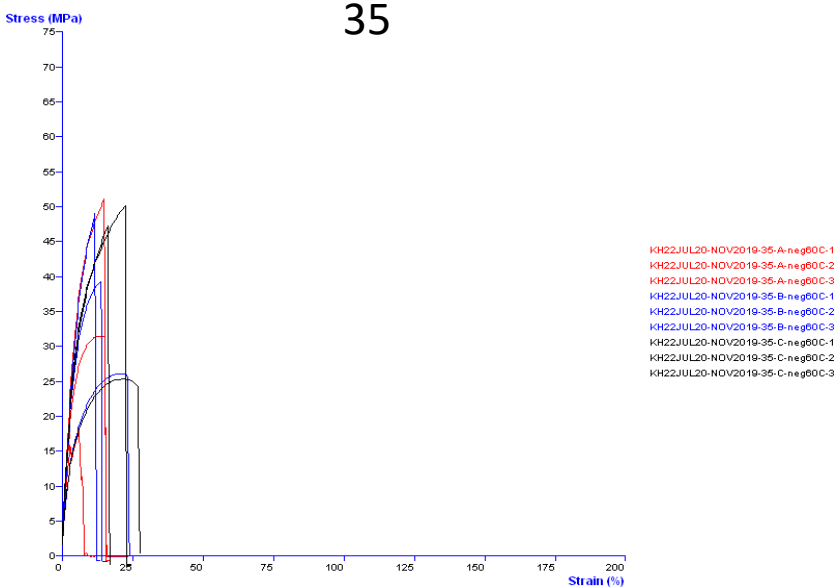
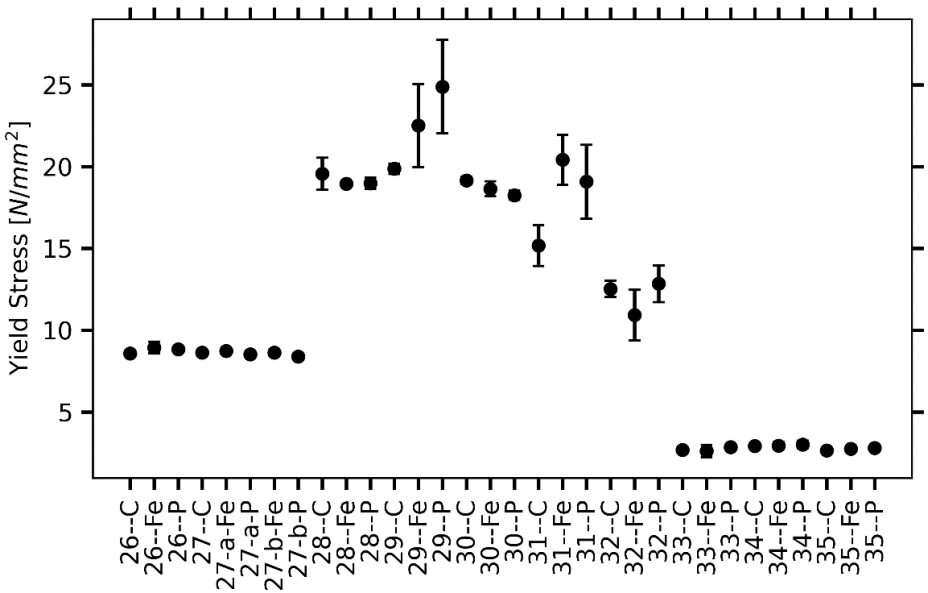
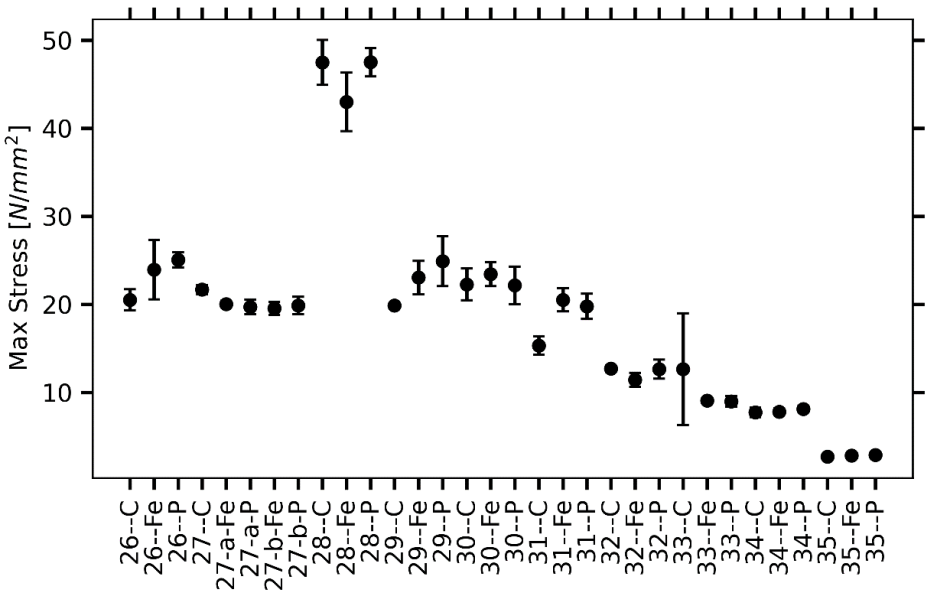
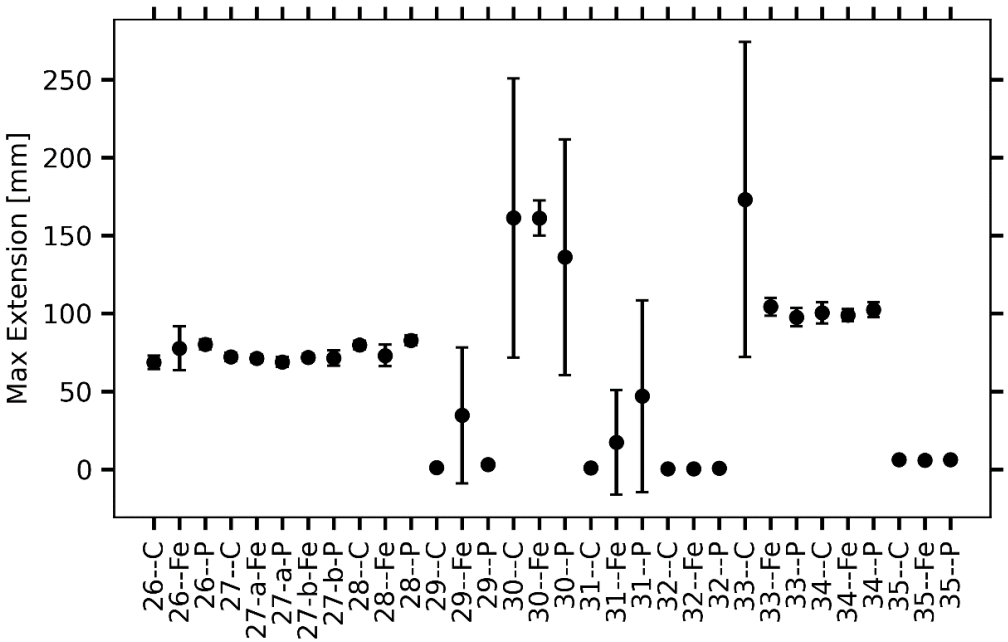
33



34



35



Mechanical Testing