H-Mat Overview: Polymers

May 27, 2020

Kevin L Simmons

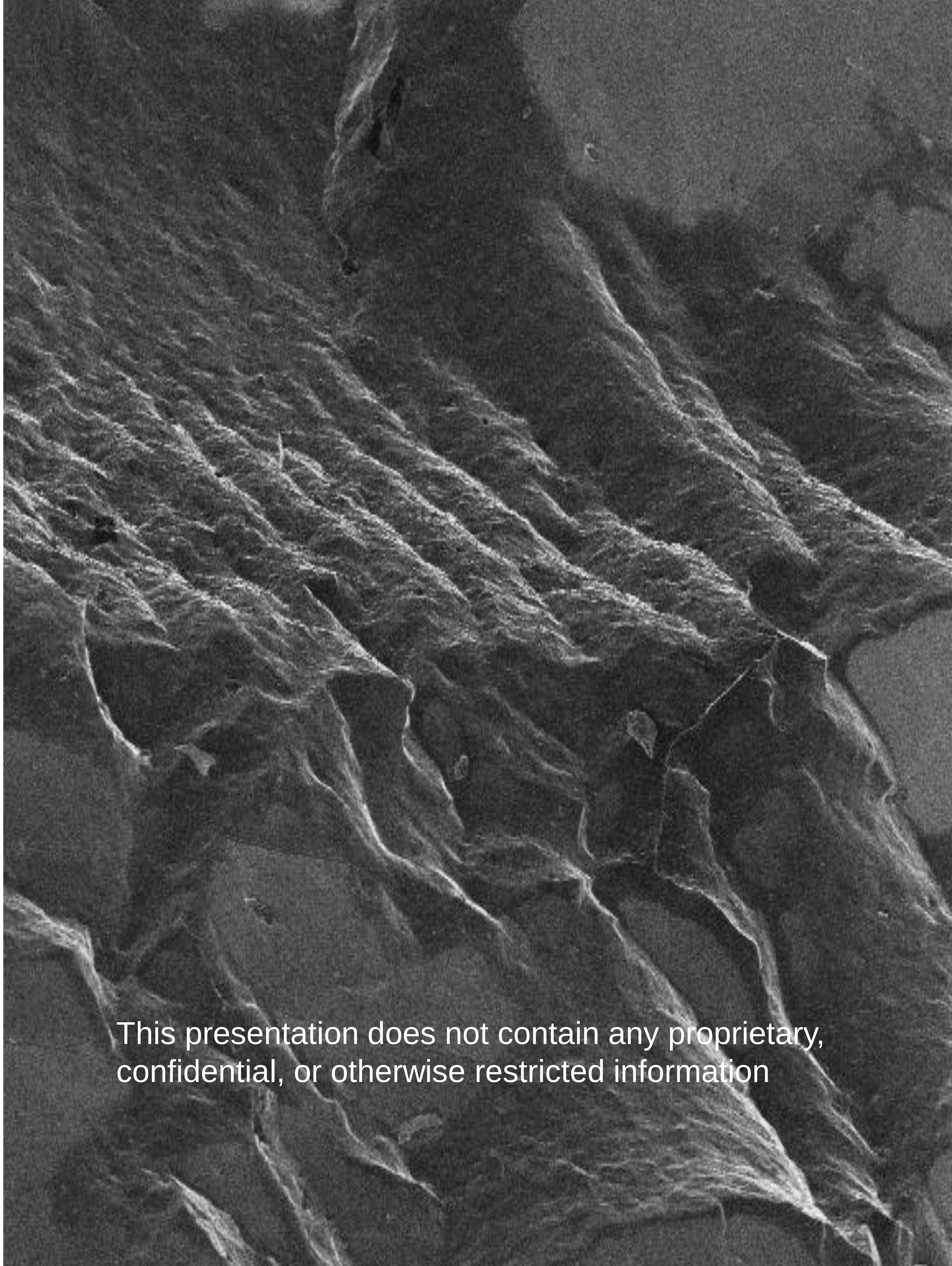
H-Mat Co-Lead Polymers

Pacific Northwest National Laboratory

2020 Annual Merit Review

SCS_026

PNNL is operated by Battelle for the U.S. Department of Energy



Pacific Northwest
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H-Mat Consortium Members

**Kevin Simmons, H-Mat Co-Lead
Polymers
H-Mat Team**

PNNL:
Wenbin Kuang
Yongsoon Shin
Sarah Burton
Kee Sung Han
Erin Barker
Yulan Li
Shank Kulkarni
Bruce Arey
Alice Dohnalkova

Industrial Collaborators:
Arlanxeo
TSE Industries
Burke Industries
Zeon Corporation
Takaishi Industry (Japan)

SNL:
Nalini Menon
Jeff Campbell
April Nissen
Bernice Mills
Mark Wilson
Amilie Frischknecht
ORNL:
Bart Smith
Amit Naskar
Larry Anovitz
SRNL:
Will James
ANL:
Hee Seok Roh
Academic Collaborator:
Kyushu Univ (Japan)

H-Mat SM
Hydrogen
Materials
Compatibility
Consortium

Pacific Northwest
NATIONAL LABORATORY

OAK RIDGE
National Laboratory

Sandia
National
Laboratories

SRNL TM

Argonne
NATIONAL
LABORATORY

Ford

Takaishi Industry Co.,Ltd.
高石工業株式会社

TSE

ARLANXEO
Performance Elastomers

KYUSHU UNIVERSITY

Burke Industries
An Engineered Polymers Company

ZEON
ZEON CORPORATION

Overview

Timeline

- Project Start Date: September 2018
- Project End Date: September 2022
  - % Completed: 30%
- Total FY19 Budget: $1149K

Planned FY20 Funding: $660K

Partners

- PNNL (H-Mat Polymer Lead)
- SNL
- ORNL
- SRNL
- ANL

Barriers

Safety, Codes, and Standards

A. Safety Data and Information: Limited Access and Availability

G. Insufficient Technical Data to Revise Standards

J. Limited Participation of Business in the Code Development Process

K. No consistent codification plan and process for synchronization of R&D and Code Development

Hydrogen Delivery

B. Reliability and Costs of Gaseous Hydrogen Compression

E. Gaseous Hydrogen Storage and Tube Trailer Delivery Costs

I. Other Fueling Site/Terminal Operations

Collaborators

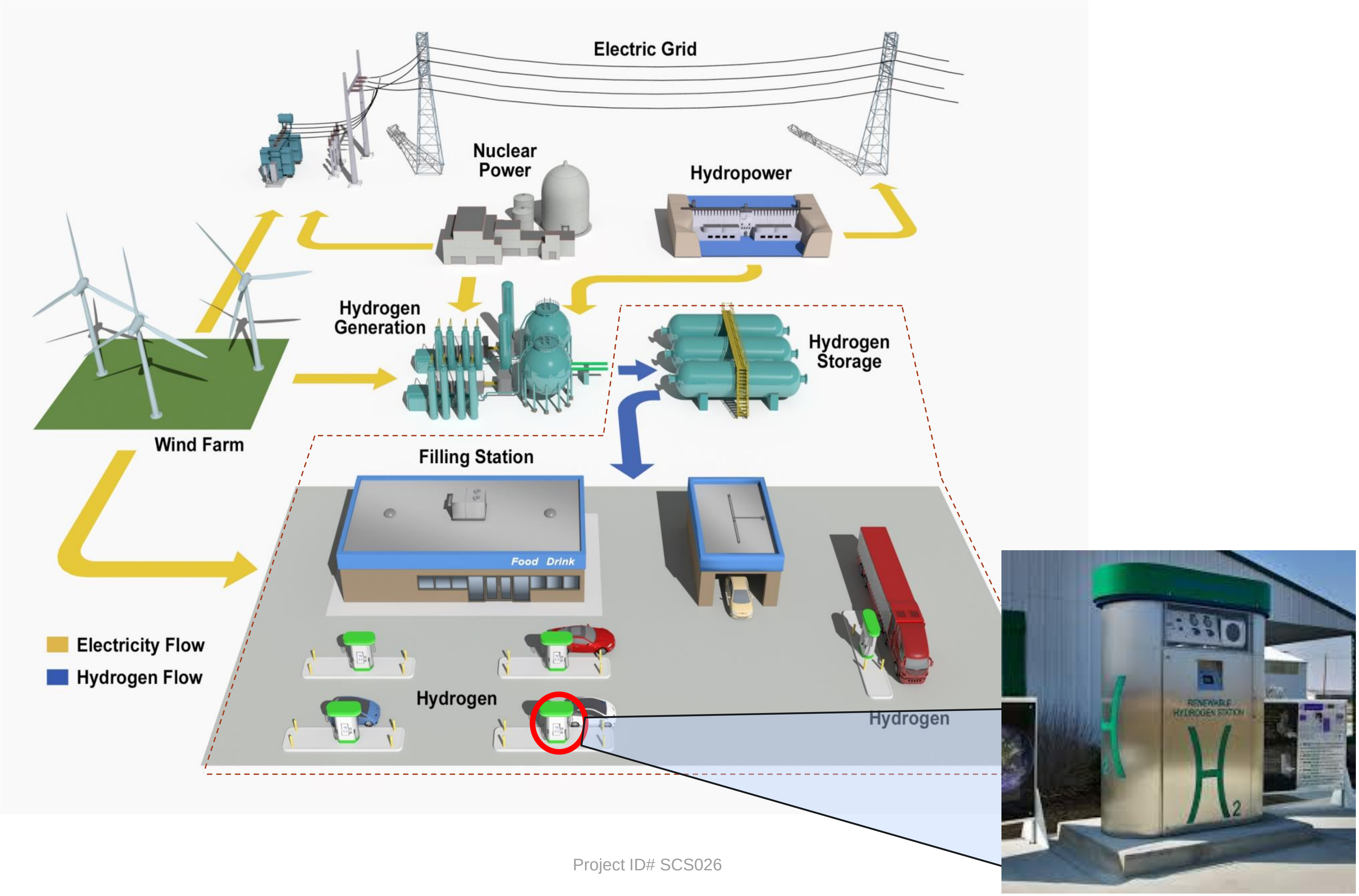
Swagelok, Takaishi Ind., Arlanxeo, Zeon, TSE, Chemours

Kyushu University (Hydrogeniuous)

H2@Scale

Polymeric materials are used throughout the hydrogen infrastructure for its sealing and flexible barrier performance

However, it’s hydrogen performance is not well understood and is a source of system failures that create an unreliable infrastructure



H-Mat Relevance

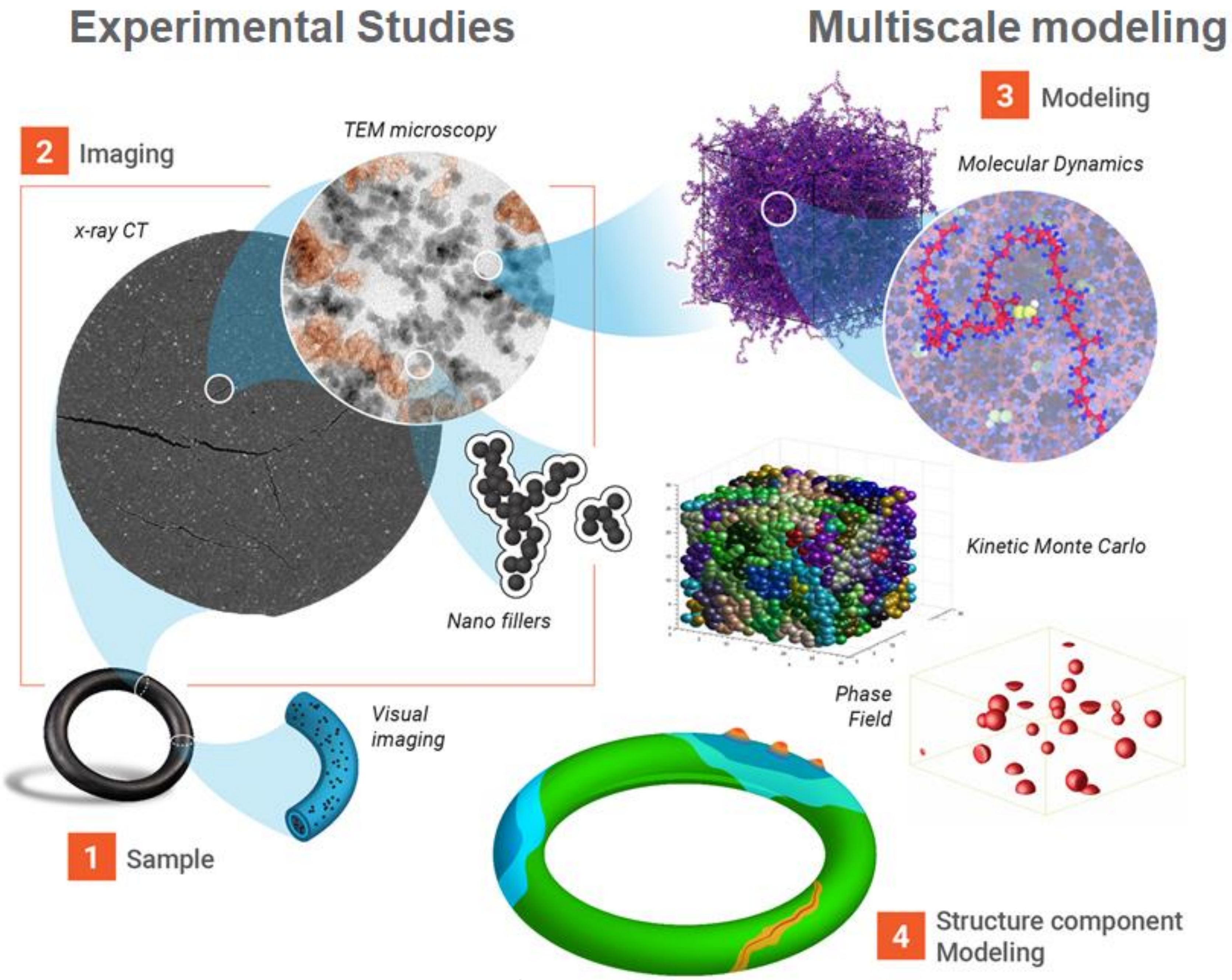
H-Mat was formed to address the hydrogen compatibility performance of materials to increase the durability of material thereby providing a more reliable and stable performance of systems in the hydrogen infrastructure

Objective: To address the challenges of hydrogen degradation by elucidating the mechanisms of hydrogen-materials interactions with the goal of providing science-based strategies to design materials (micro)structures and morphology with improved resistance to hydrogen degradation.

Task	Relevance and Objectives
Mechanisms of hydrogen-induced degradation of polymers	Quantify the hydrogen pressure-temperature-time-damage relationships of polymers with controlled structure and morphology (to inform models of hydrogen-induced degradation of polymers
Computational multiscale modeling	Develop material damage models of process-structure-property performance with the aim of motivating materials formulations that are less sensitive to hydrogen-induced damage
Hydrogen-resistant polymeric formulations	Discover modified and new materials systems that improve hydrogen compatibility that will increase the reliability of materials and components in hydrogen infrastructure
Materials for cryogenic hydrogen service	Identify materials for cryo-compressed hydrogen storage onboard vehicles, and develop key technical metrics for viable structural materials in this application

Overall Approach

H-Mat Combines Unique Experimental, Analytical, and Computational Capabilities at the National Labs

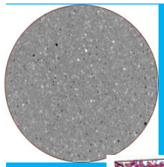


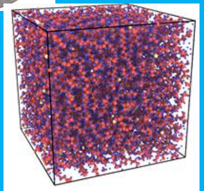
Collaborations

H-Mat Team Multidisciplinary Collaboration with Unique Capabilities in Modeling and Experimentation


Compression set test for elastomers


Density measurements set-up


Xray-CT


Atomistic modeling

Sandia National Laboratories


Pressure cycling manifold – sample preparation

ARLANXEO Performance Elastomers

TSE

ZEON ZEON CORPORATION

Burke Industries An Engineered Polymers Company

Takaishi Industry Co.,Ltd. 高石工業株式会社

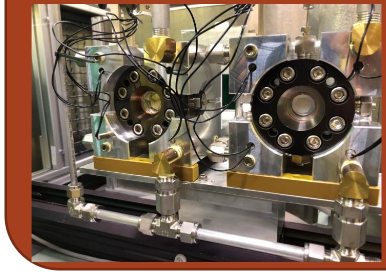
Industrial collaborators

KYUSHU UNIVERSITY

International Collaborator
Information sharing with H-Mat Team

- **Understanding H2 material effects**
- **Model materials**
- **Material modification**
- **New formulation**
- **Information dissemination**

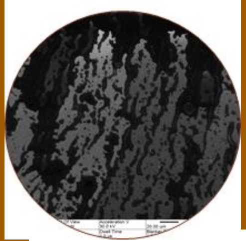
uSANS/uSAXS



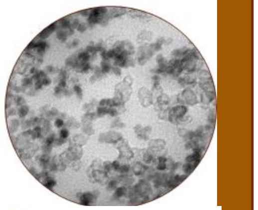
OAK RIDGE National Laboratory

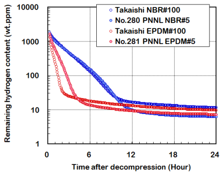
Pacific Northwest NATIONAL LABORATORY

HeIM

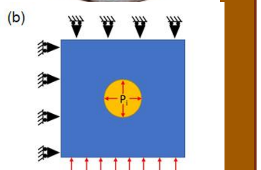


High-Res TEM



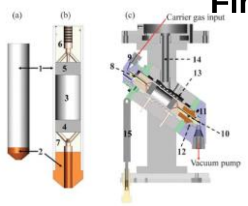


Hydrogen transport properties in materials

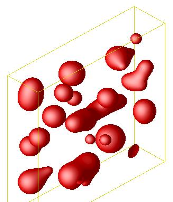


Finite element


PNNL high-pressure system



High-pressure NMR


Phase-field

Accomplishment FY19

Multiscale Modeling & Experiment Summary

Multiscale modeling:

- Atomistic Modeling
  - Provided insight as to the failure mechanisms with chemical specificity at the atomistic length scale
- Phase Field Model
  - Developed model for gas bubble formation & coalescing during rapid gas decompression at a meso length scale
- Component Scale Modeling
  - Developing a failure predictive tool for components of interest at the component length scale, to aid in new materials formulation & design for improved resistance to hydrogen exposure

Experimental:

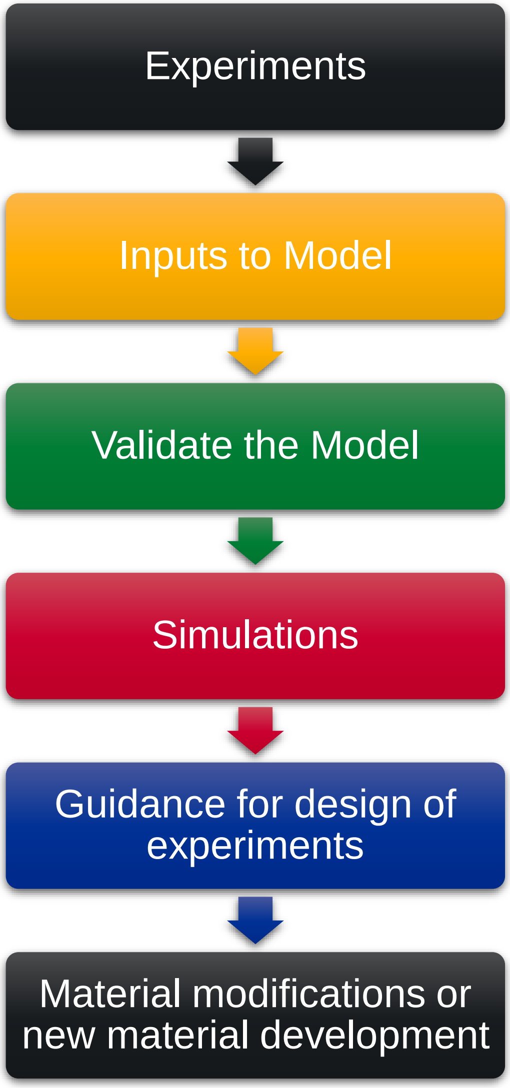
- PNNL capabilities development
  - *In-situ* Dynamic Mechanical Analysis (*in-situ* DMA) to quantify the property-temperature-pressure-time relationship
  - Hydrogen Permeation Tester to quantify transport properties of hydrogen in polymers
- Advanced characterization techniques
  - Helium Ion Microscope (HeIM) discovered chemical & morphological changes in polymer induced by high pressure hydrogen
  - Transmission Electron Microscope (TEM) unveiled filler distribution within the materials and chemical changes occurred around Zinc particles with Energy Dispersive X-ray Spectroscopy (EDS)
  - Nuclear Magnetic Resonance (NMR) indicated hydrogen diffused and accumulated within the material and provided information to calculate diffusion coefficients

Development of Datahub for disseminating information to the hydrogen community

H-Mat Modeling and Experimental Workflow

- Experiments will provide following for the model:
  - Visual observations
  - Material properties
  - Topography of cavities and/or bubbles
  - Validation data

- Simulations will provide following for experiments:
  - Optimum parameters
  - Trends and what to expect
- Develop guidance to material modifications and new material developments for improved hydrogen material compatibility



Experiments

Inputs to Model

Validate the Model

Simulations

Guidance for design of experiments

Material modifications or new material development

Progress and Accomplishments

Atomistic modeling: Understanding the effects of crosslink density dependence during decompression

SUCCESSES:

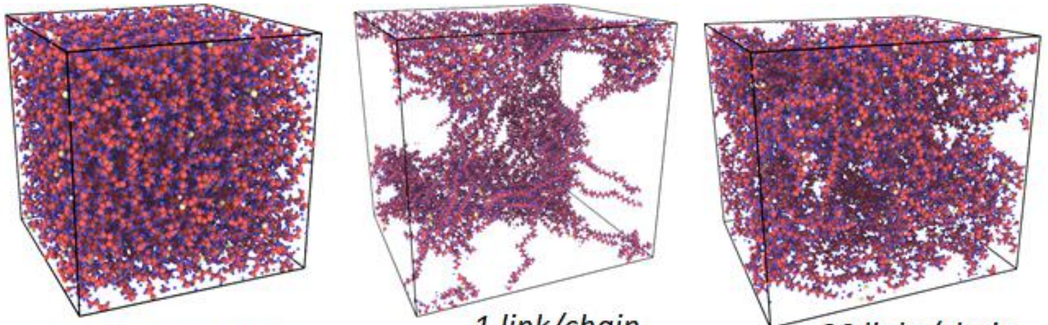
- Improved accuracy of all-atom material representation
- Developed preprocessing method to crosslink polymers at desired densities
- Implemented method to assure correct gas solubility-pressure relationships during H2 gas insertion.

METHOD:

- Molecular dynamics simulations are performed for all-atom representations of EPDM
- Various degrees of crosslinking are imposed in initial configurations
- H2 gas is inserted into EPDM configurations at various pressures
- Simulations model rapid decompression to I atm

SIGNIFICANCE:

- Structural rearrangement of polymer chains is slowed with increased content of crosslinks
- Suggests a composition with increased percentage NBR could reduce H2 susceptibility to bubble formation



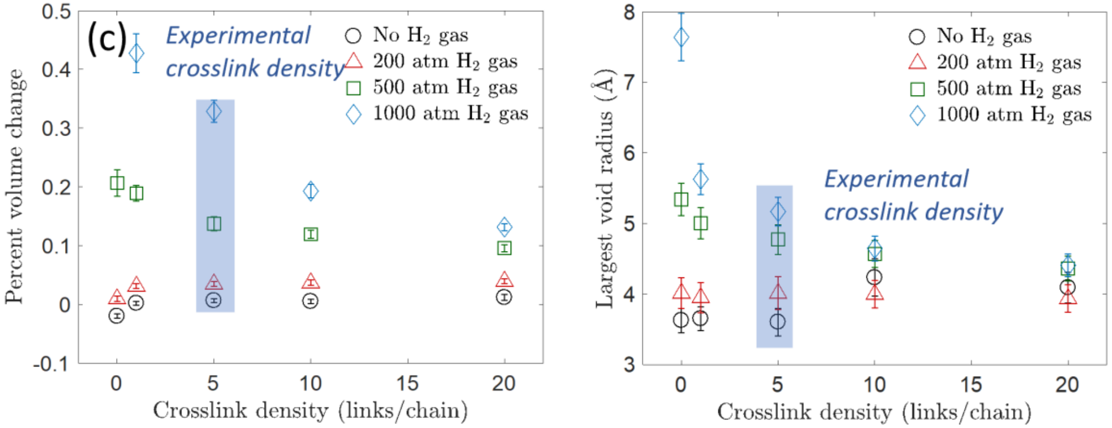
Initial 1 g/cc EPDM

1 link/chain

20 links/chain

After decompression

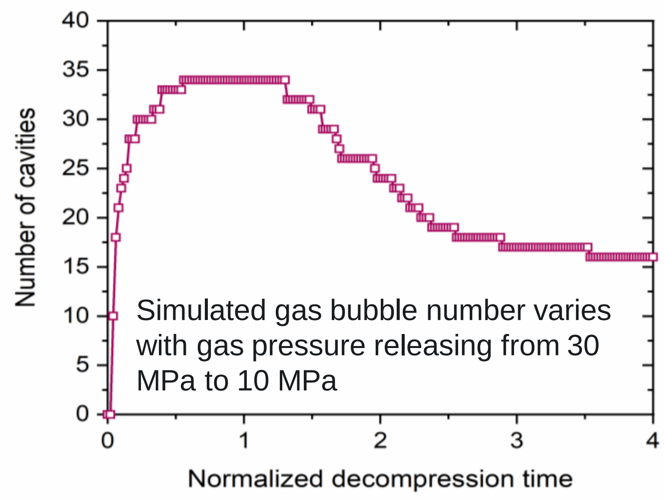
***(a)** Initial configurations at 1g/cc are crosslinked and pressurized with H2 gas to desired content. Hydrogen not shown for clarity. **(b)** Decompression simulations are performed till equilibrated at 1 atm. **(c)** Structural changes are then assessed.*



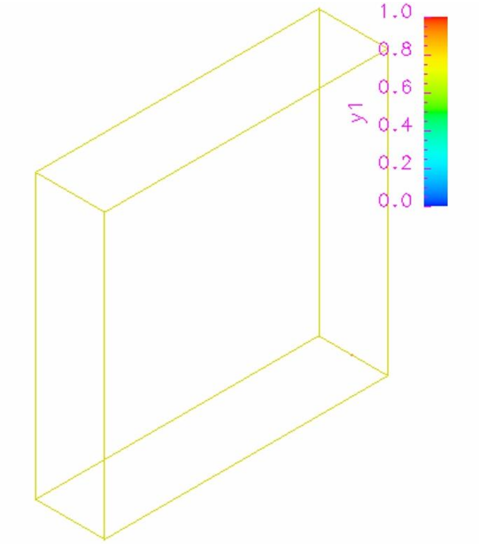
Progress and Accomplishments

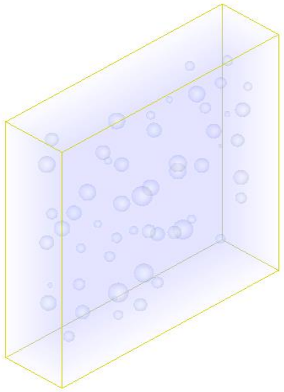
Phase-field modeling: Model development of cavitation in rubbers during decompression

- Cavitation occurs during decompression after rubbers exposed to high-pressure H2 gas
- Existing free volume in rubbers affects the cavity evolution as well as material performance
- A phase-field model has been developed to simulate the thermodynamic and kinetic process of cavity evolution, and predict the effect of structural defects and decompression rate on cavity nucleation, growth and coalesce kinetics

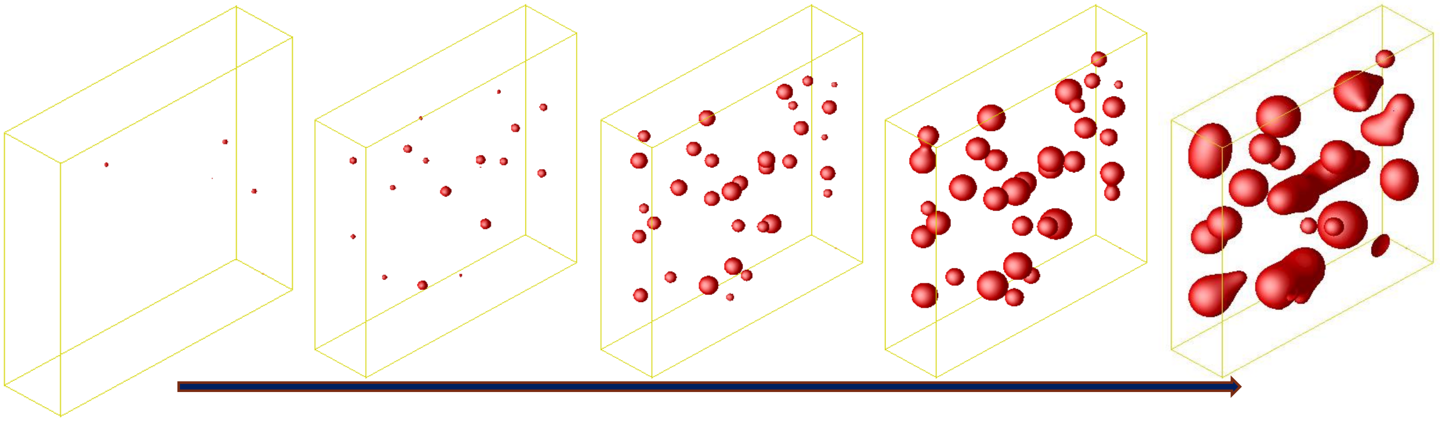


Simulated gas bubble number varies with gas pressure releasing from 30 MPa to 10 MPa





Distribution of initial free volume



Simulated cavity structure evolution during decompression: first gas bubbles nucleate from existing free volumes, then grow and coalesce, finally shrink.

Progress and Accomplishments

Finite element modeling: Coupled diffusion-deformation analysis to predict damage initiation and propagation in polymer

- A continuum mechanics-based deformation model to predict damage evolution during pressurization and depressurization cycles.
- The diffusion analysis is performed first and output from diffusion analysis provides input to deformation analysis.
- The polymer is modeled as hyper-elastic material behavior using tensile test response.
- Model can be easily extended to include multiple cavities and filler particles.
- Simple and computationally inexpensive.

Pressure profiles taken to be same as H_2 conc. obtained from diffusion analysis.

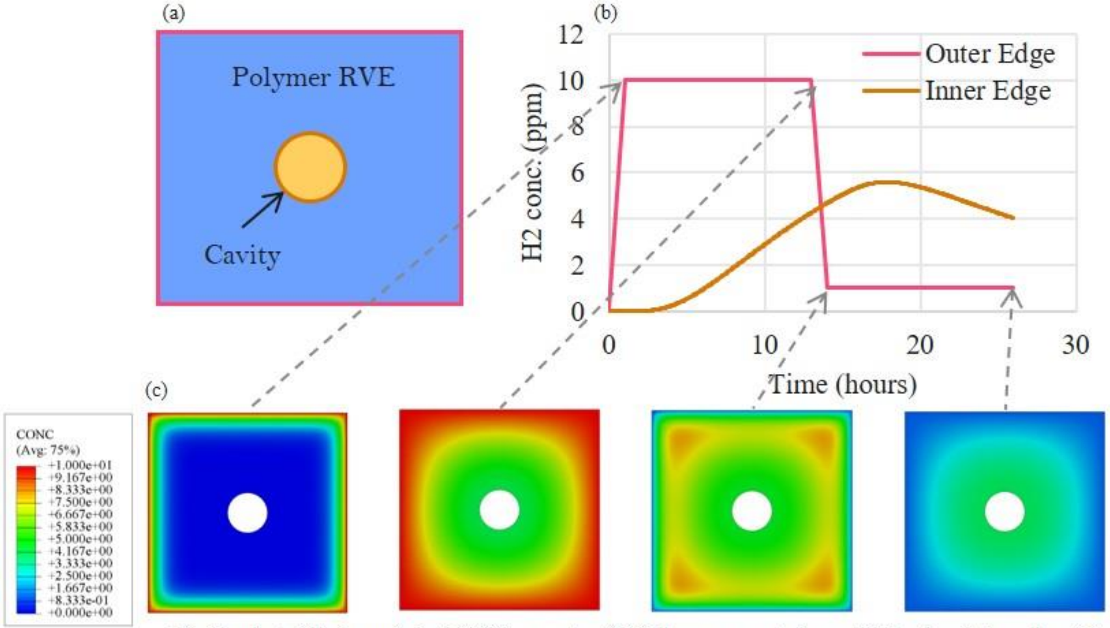


Fig: Transient diffusion analysis. (a) RVE geometry , (b) Hydrogen concentration on RVE and cavity boundary, (c) hydrogen concentration at different time in pressurization and depressurization cycle.

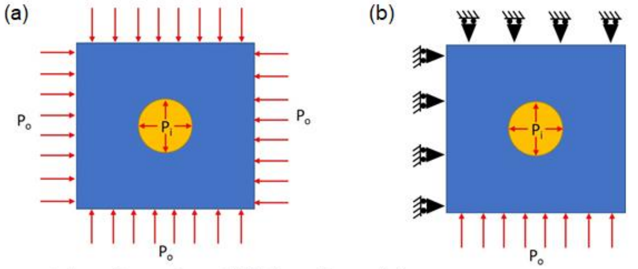
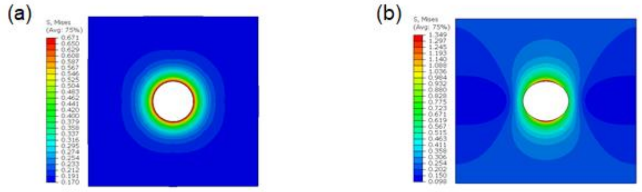


Fig: Representative volume element (RVE) used for modeling the polymeric component with cases of: (a) Free boundary conditions and (b) Mixed boundary conditions.



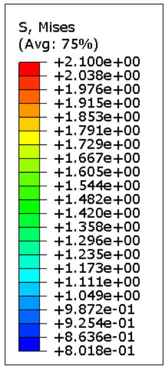


Fig: Stress distribution around the cavity after depressurization in the case of: (a) Free boundary conditions and (b) Mixed boundary conditions.

Multiple cavities with different sizes

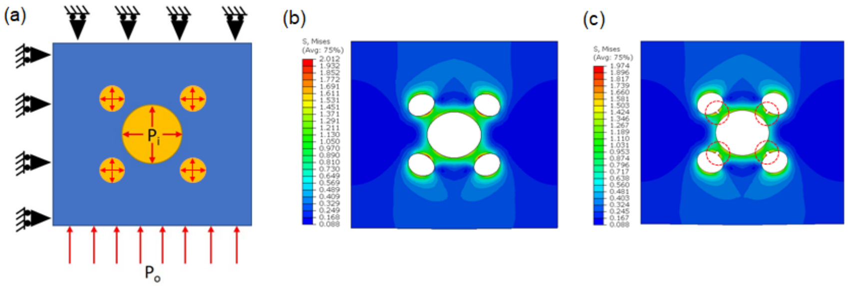


Fig: Interaction of multiple cavities. (a) RVE geometry with BC, (b) Stress distribution, and (c) Damage initiation.

Effect of filler particles (Silica, carbon black etc.)

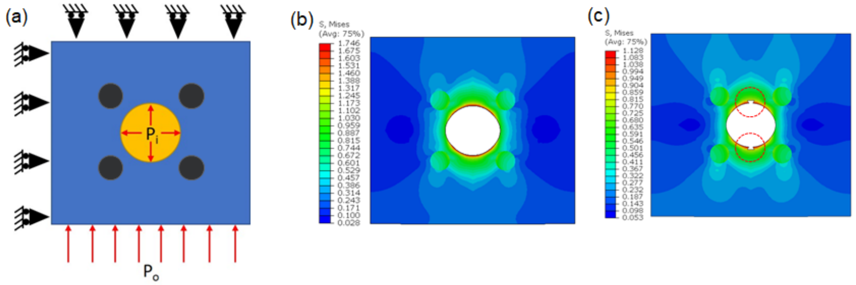


Fig: Effect of filler particles. (a) RVE geometry with BC, (b) Stress distribution, and (c) Damage initiation.

Progress and Accomplishments

Development of Model Material Compounds for Experimental Studies and Industry Collaboration Materials

- **Elastomer**
  - Model Materials:
    - EPDM: E1 (no filler, no plasticizer), E2 (p, nf), E5 (f, p), E6 (f, np)
    - NBR: N1 (nf, np), E2 (p, nf), E5 (f, p), E6 (f, np)
    - FKM: % Flourine (need to update)

  - Other Formulations:
    - HNBR (Burke materials, Zeon materials, Arlanxeo materials)
    - EVM (Arlanxeo materials)
    - CR (Arlanxeo materials)
    - PU (TSE materials)

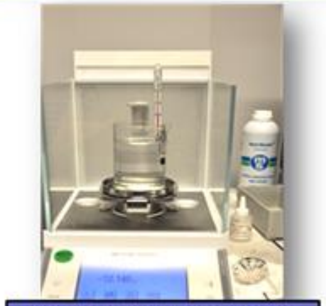
- **Thermoplastics**
  - POM
  - PTFE
  - Nylon 6,6
  - PEEK
  - Nylon 11
  - HDPE

Significant interest and support from industry with materials and oil and gas industry experience with high pressure

Progress and Accomplishment

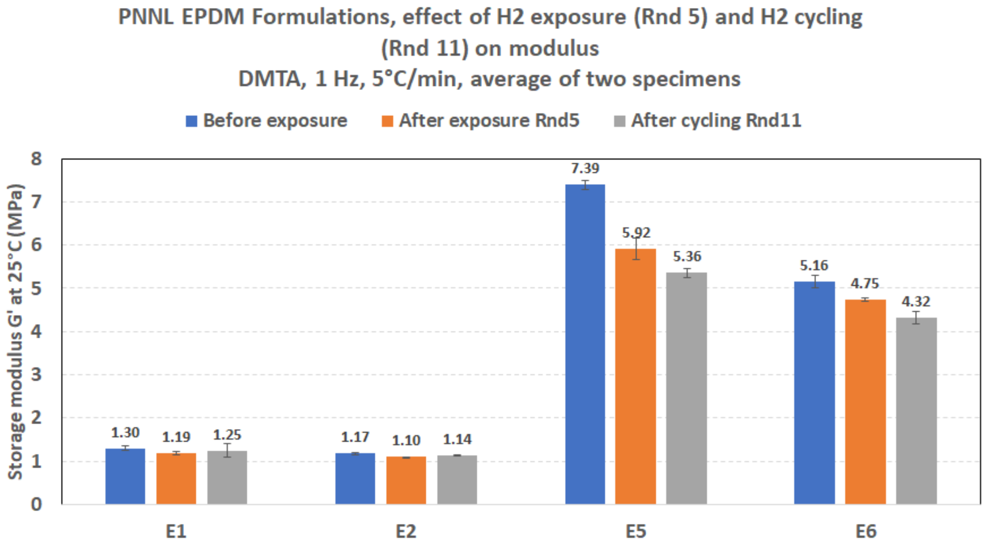
Ex-situ Characterization – physical, mechanical, and structural properties

Compression set test for elastomers



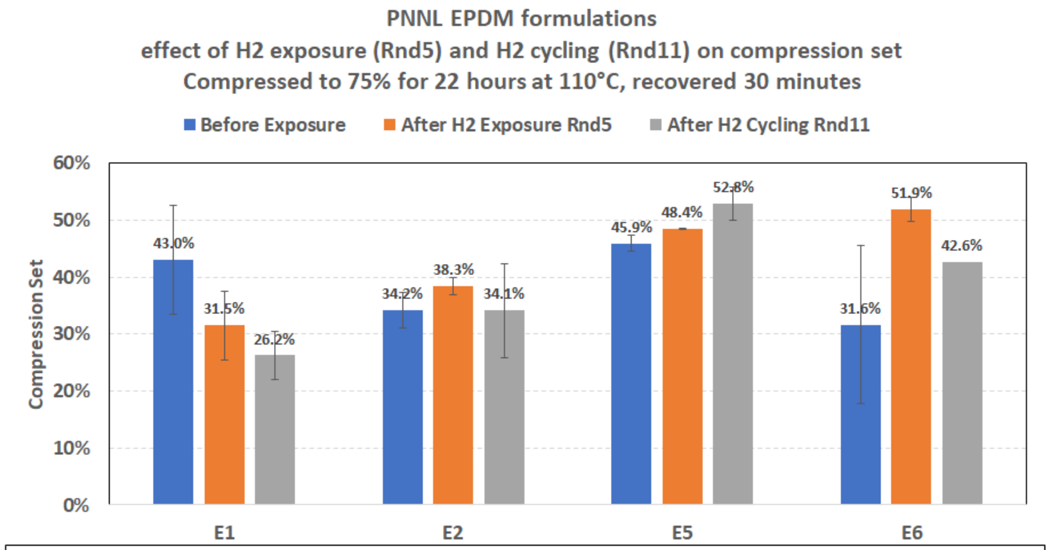
Density measurements set-up

All samples treated in hydrogen at 90 MPa



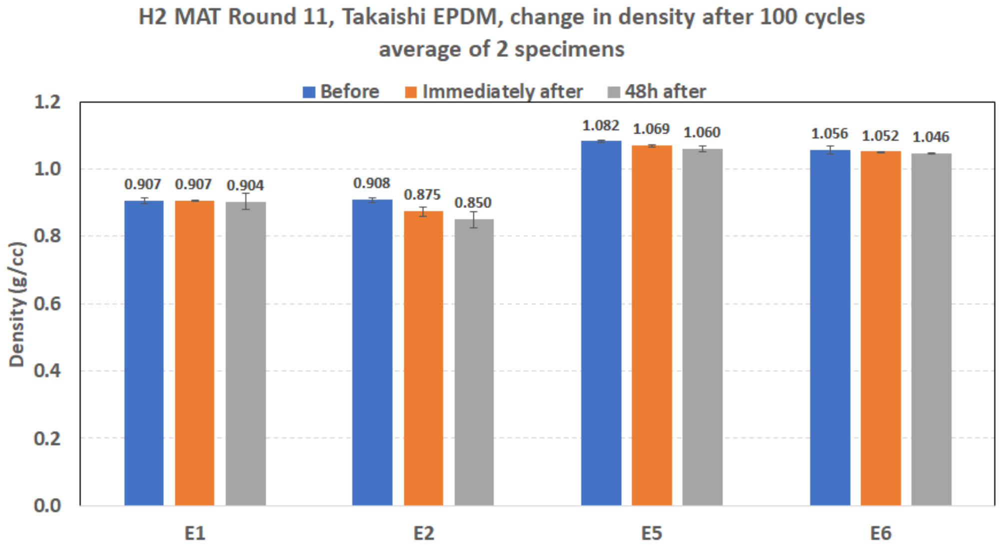
PNNL EPDM Formulations, effect of H2 exposure (Rnd 5) and H2 cycling (Rnd 11) on modulus
DMTA, 1 Hz, 5°C/min, average of two specimens

Significant change in storage modulus indicates possible plasticization of EPDM E5 (and E6) and influence due absorption/retention of H_2 by fillers



PNNL EPDM formulations
effect of H2 exposure (Rnd5) and H2 cycling (Rnd11) on compression set
Compressed to 75% for 22 hours at 110°C, recovered 30 minutes

Compression set for E5 increases upon H2 exposure for both static and cycling modes – possible plasticization of matrix and/or retention of H_2 by fillers



H2 MAT Round 11, Takaishi EPDM, change in density after 100 cycles
average of 2 specimens

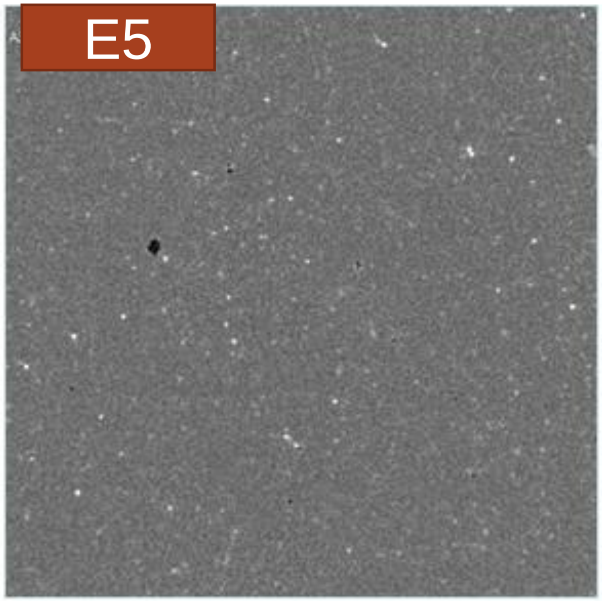
Densities of EPDM formulations change insignificantly upon H2 exposure – minimal physical swelling due to tight network, high crosslink density and less free volume

Progress and Accomplishment

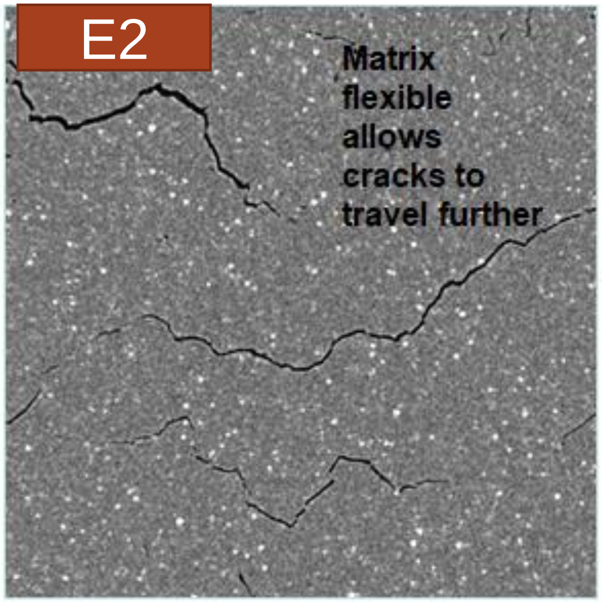
Post-mortem X-ray Computed Tomography

E1, E2, E5 and E6 after week-long static exposure of 90 MPa H_2

E5



E2

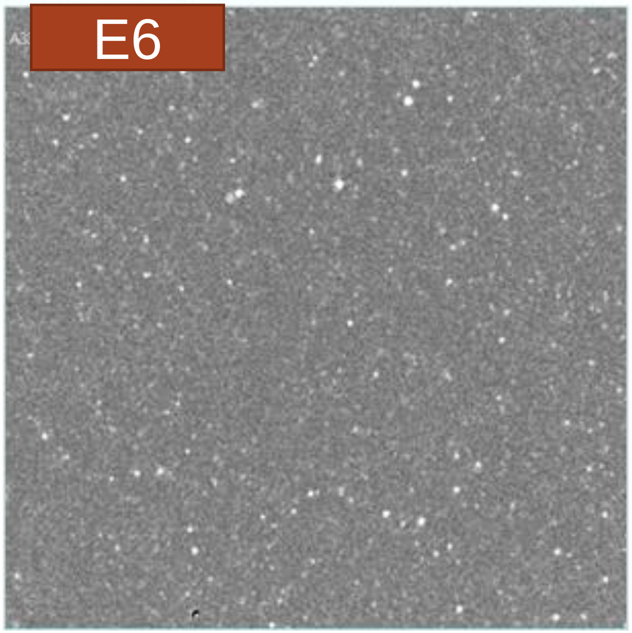


Matrix flexible allows cracks to travel further

For custom EPDM formulations, presence of plasticizer changed the nature of cracks formed.

For custom EPDM formulations, addition of C and SiO_2 fillers helps mitigate crack formation.

E6



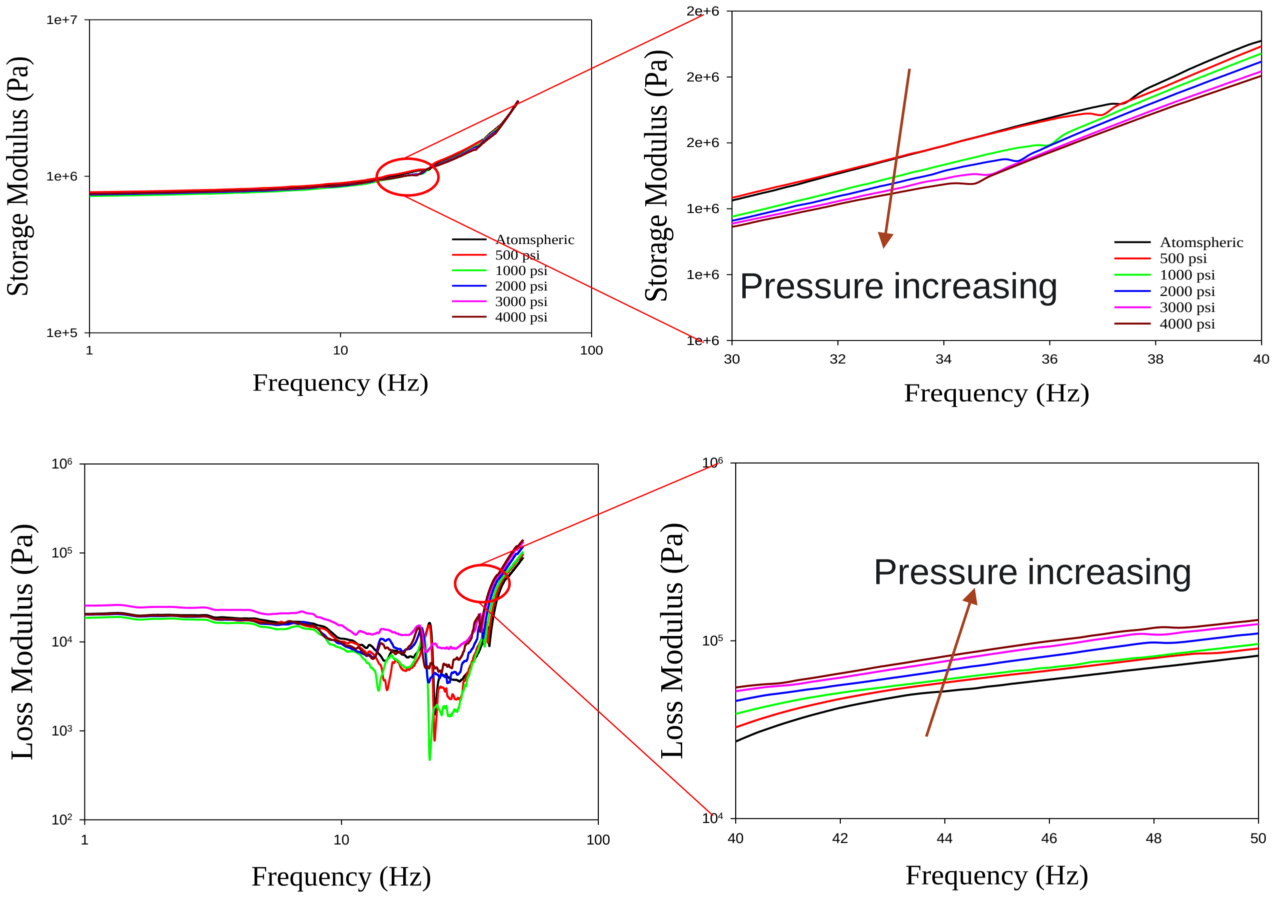
E1

Matrix very brittle; short cracks

Progress and Accomplishment

In-situ DMA: Pressure Effect of Helium on E1

Frequency Sweep @RT



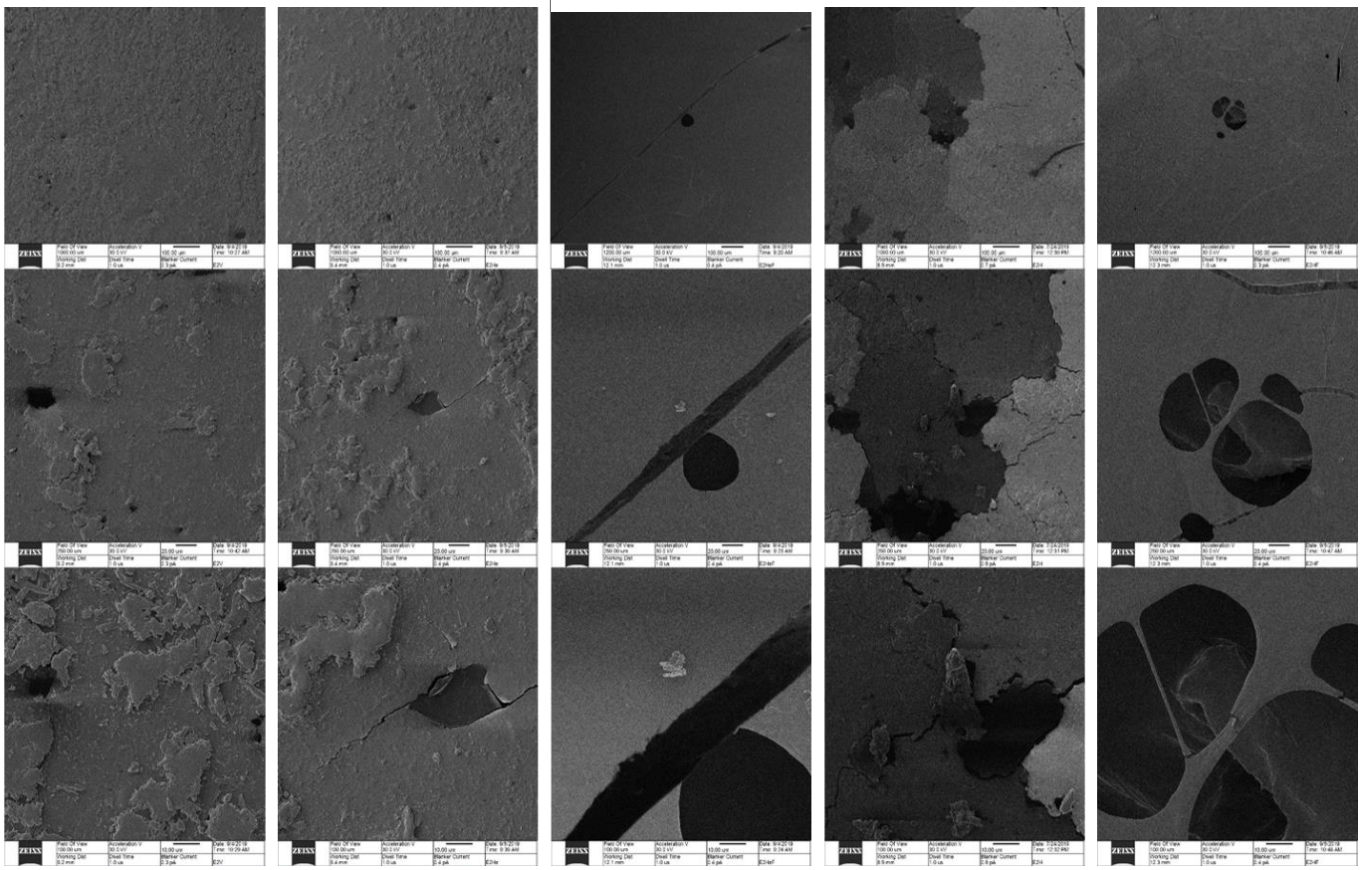
- Step-pressurization (atmospheric, 500, 1000, 2000, 3000, 4000 psi)

	E' at w=34	**Δ (based on atmospheric condition)**
Atmospheric	1.59 MPa	0%
500 psi	1.59 Mpa	0%
1000 psi	1.53 MPa	-3.8%
2000 psi	1.51 MPa	-5.0%
3000 psi	1.49 MPa	-6.3%
4000 psi	1.47 MPa	-7.5%

- Storage modulus (deformation resistance) reduces with pressure increasing due to pseudo-plasticization
- Pressure effect more significant at high frequencies
- Loss modulus (damping/irreversible deformation) increases with pressure increasing
- Combining storage and loss modulus data suggests that elastomer deteriorates in mechanical performance under pressure and level of deterioration depends on pressure (gas?)

Progress and Accomplishment

Helium Ion Microscopy of E2 – Hydrogen effect vs. Pressure effect



As rec’d surface before exposure

Surface morphology after 27.6 MPa He

Cryo fracture morphology after 27.6 MPa He

Surface morphology after 27.6 MPa H_2

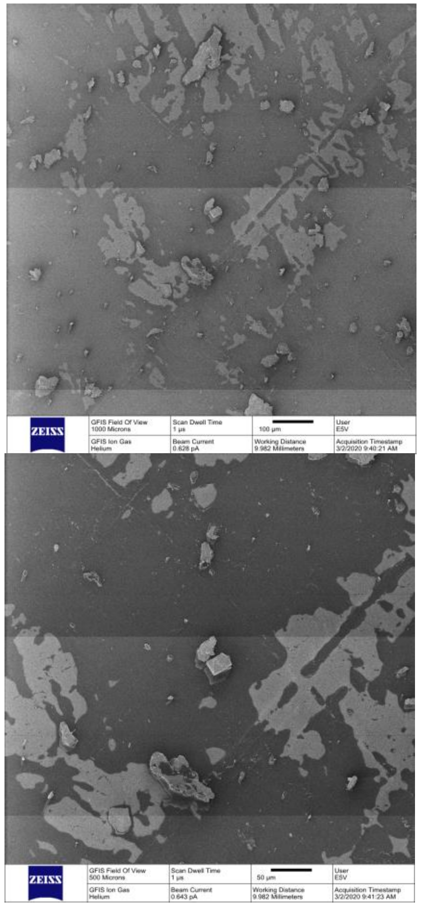
Cryo fracture morphology after 27.6 MPa H_2

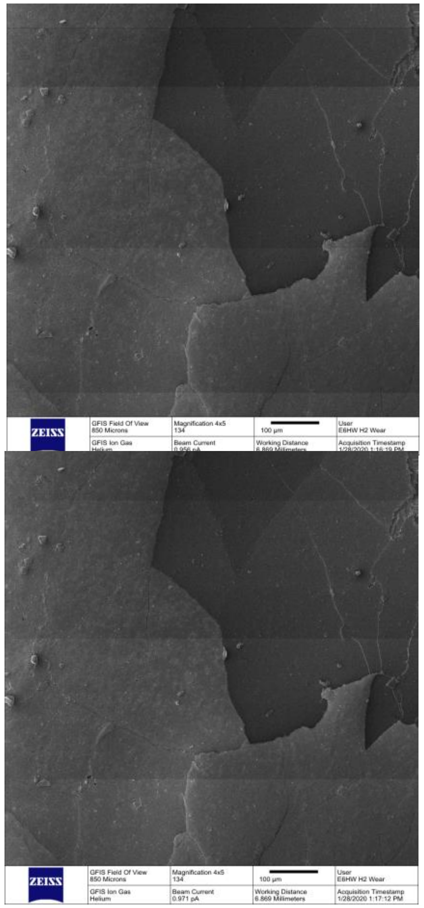
- Very little change between the as rec’d surface and the high pressure helium surface morphology
- Exposure to high-pressure hydrogen caused formation of micro-cracks and voids, and that phase separation of the plasticizer from the polymer
- Previous work with HeIM with ToF-SIMS show the dark regions to be plasticizer

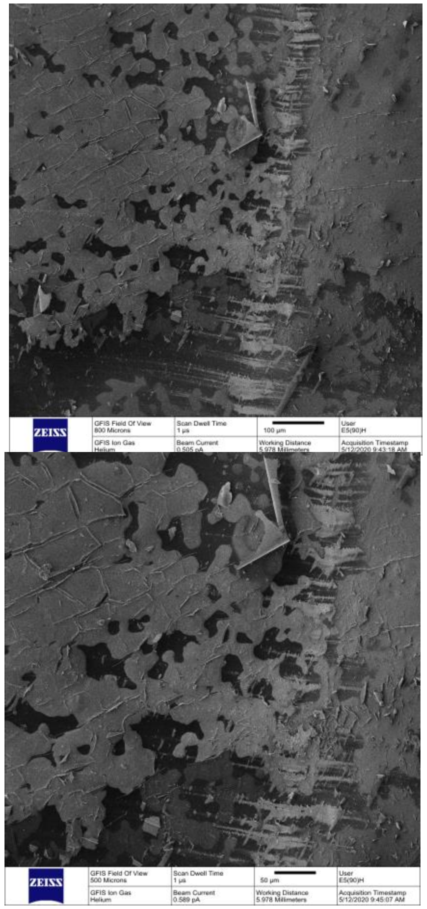
Progress and Accomplishment

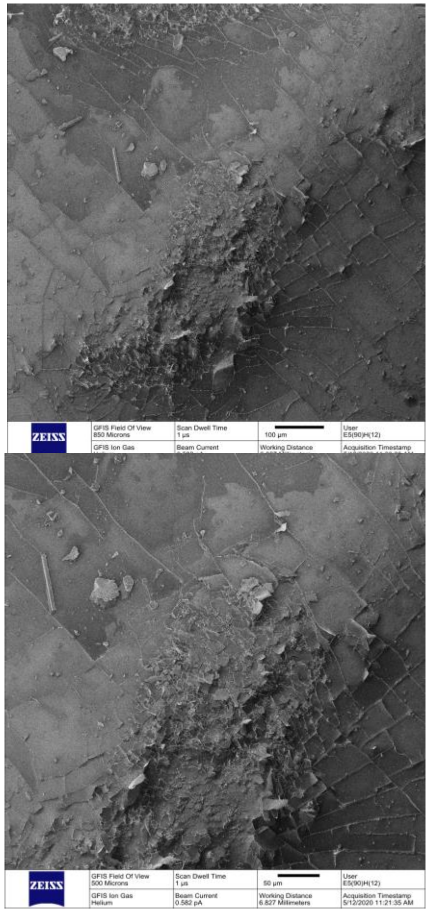
Helium Ion Microscopy of E5 – high pressure effects & effects of pressure cycling

SNL's unique high-pressure cycling system prepared certain samples for PNNL


As rec'd surface before exposure


Surface morphology after 27.6 MPa H_2


Surface morphology after 90 MPa H_2

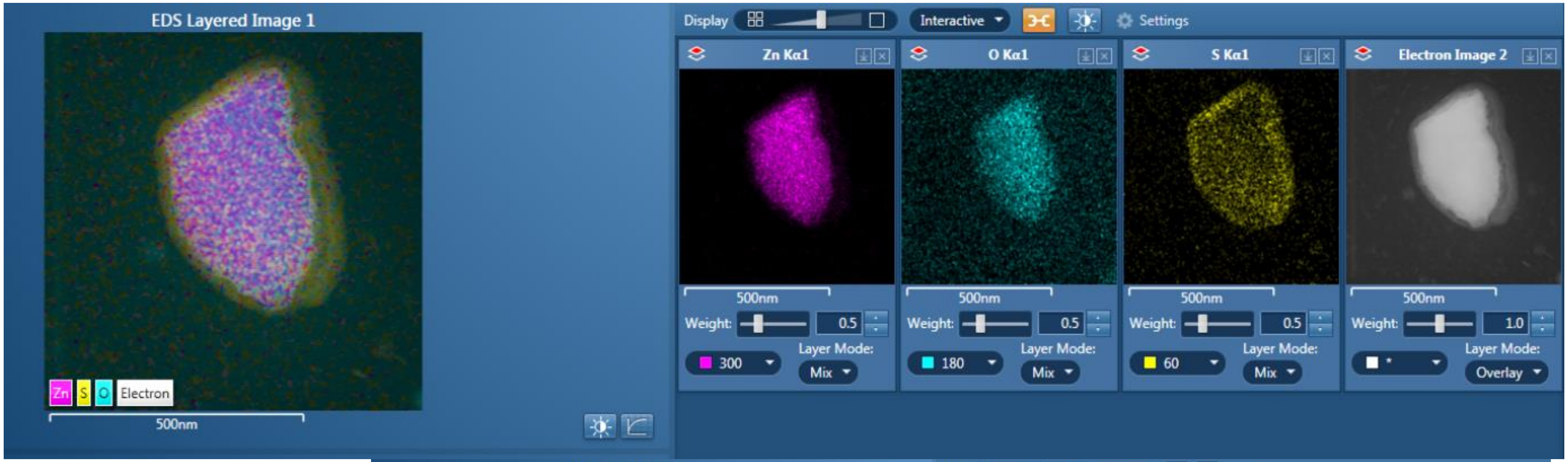

Surface morphology after 90 MPa H_2 for 12 cycles

- Noticeable surface morphology change (cracks) after exposed to 27.6 MPa H_2
- After a single exposure to 90 MPa H_2, significant phase separation and surface crack formation observed
- When exposed to 90 MPa H_2 for 12 consecutive cycles, polymer altered dramatically by forming fissures and protrusions at the surface

Progress and Accomplishments

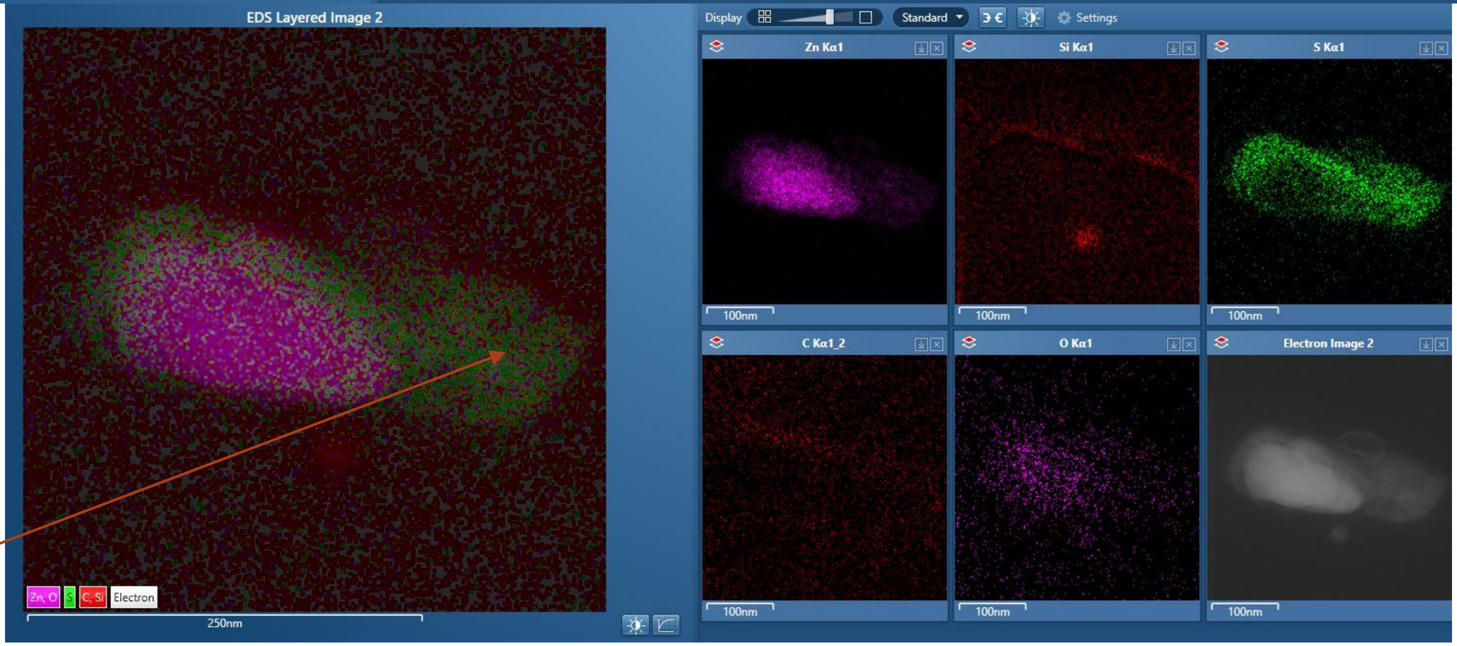
HAADF-STEM image with EDS analysis – hydrogen effects on Zinc particle and interface

E2 – 27.6 MPa __helium__ exposed



Zn Kα1 – Weight: 0.5 – 300 – Layer Mode: Mix
O Kα1 – Weight: 0.5 – 180 – Layer Mode: Mix
S Kα1 – Weight: 0.5 – 60 – Layer Mode: Mix
Electron Image 2 – Weight: 1.0 – Layer Mode: Overlay

***E2 – 27.6 MPa __hydrogen__ exposed samples** increase sulfur regions around ZnO particles*



Sulfur in the grey areas around the particle, the ZnO formed ZnS through processing

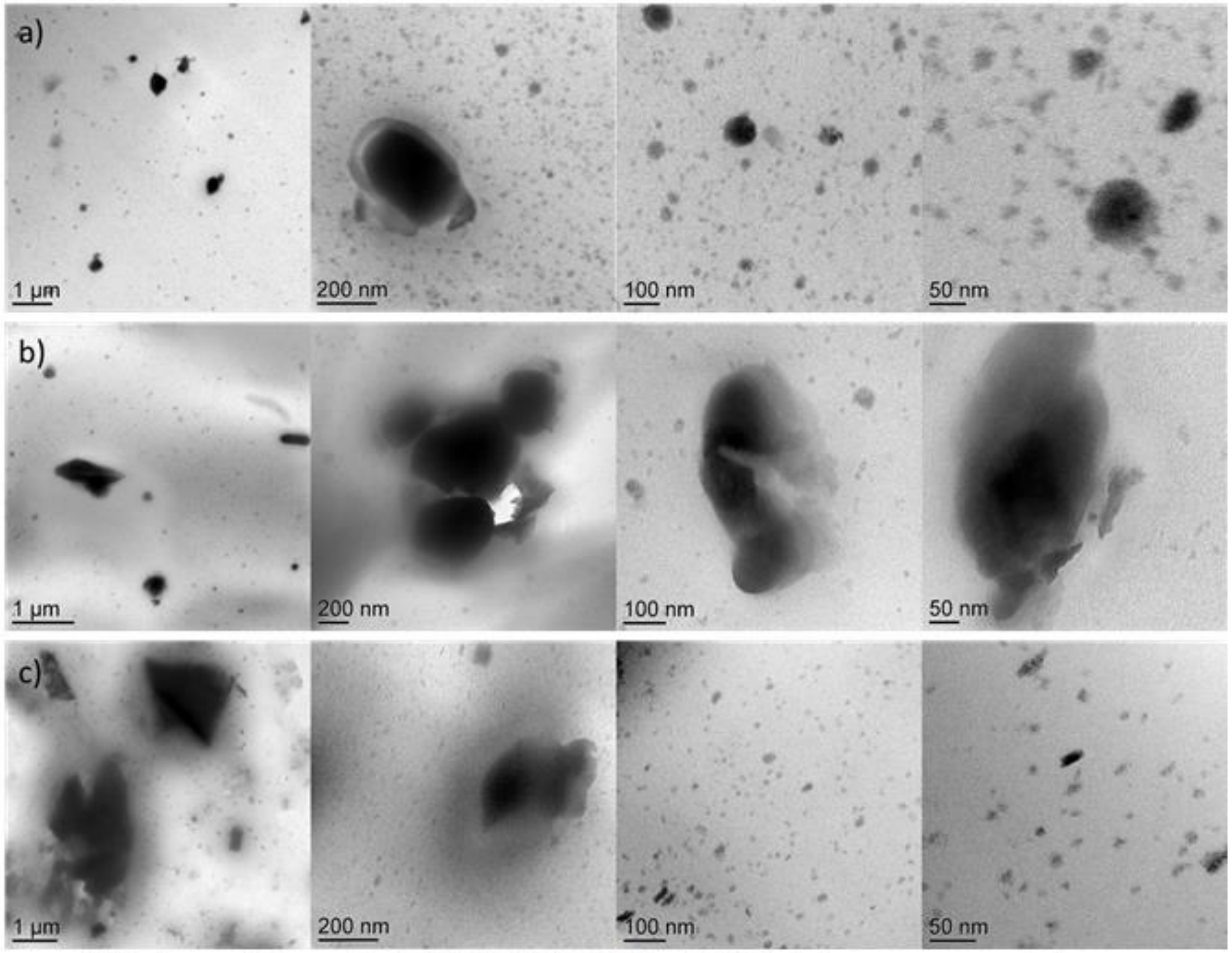
Progress and Accomplishments

Transmission Electron Microscopy of E2 (plasticizer only)

Pre (as rec'd)

Post (28 MPa He for 24 h)

Post (28 MPa H_2 for 24 h)

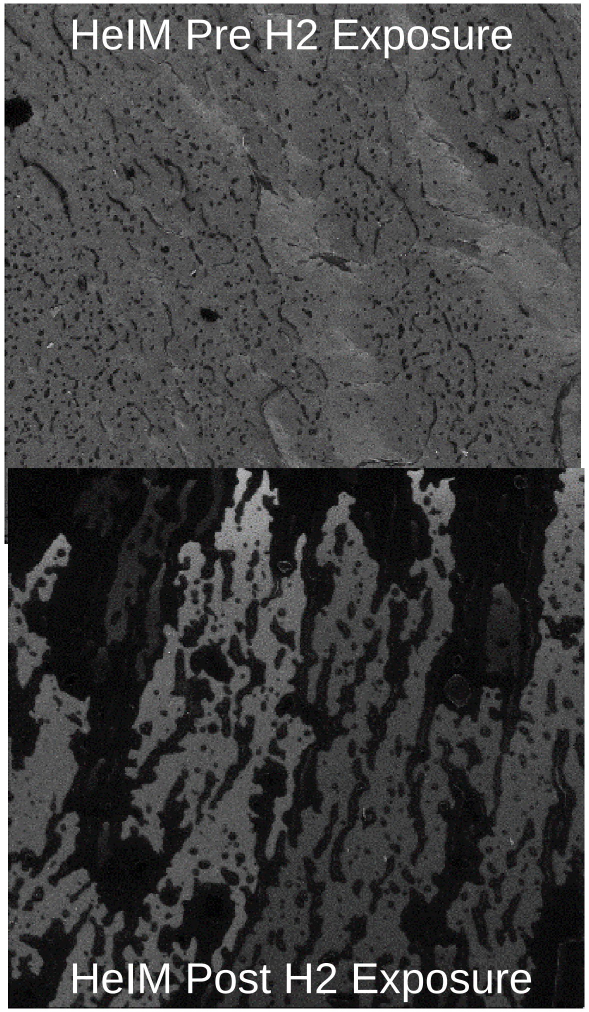


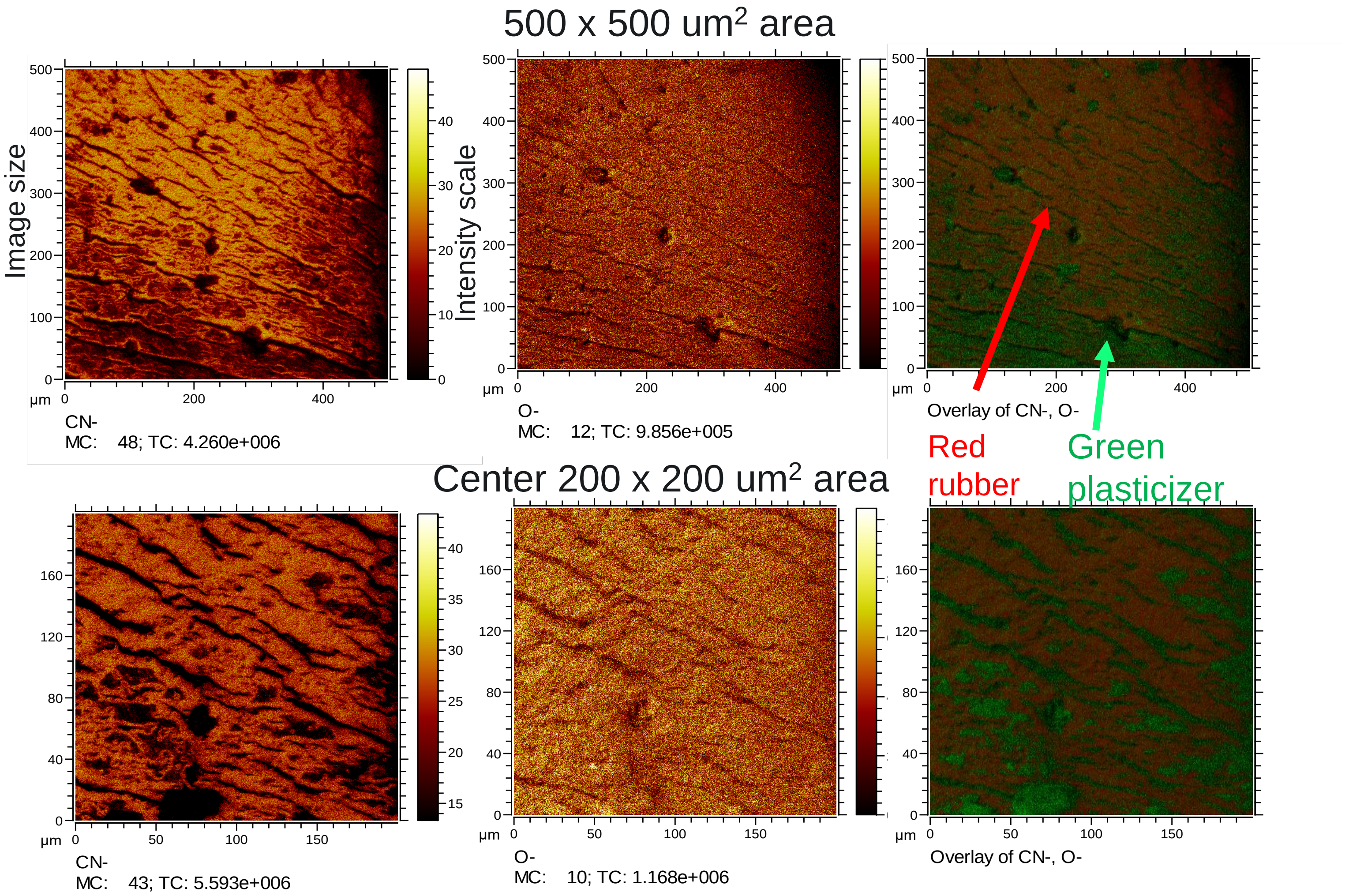
TEM images of a) as-received EPDM #2, b) EPDM #2 post 28MPa hydrogen exposure, and c) EPDM #2 post 28MPa helium exposure.

- Crack propagates after exposure to high pressure hydrogen
- Dense elements migrate towards the crack region after exposure to hydrogen
- Increased in smaller sulfur particles after H2 exposure

Progress and Accomplishments

Cryo Fractured N2 HeIM and Time of Flight Secondary Ion Microscopy (TOF-SIMS) - hydrogen effects on morphology and local chemistry





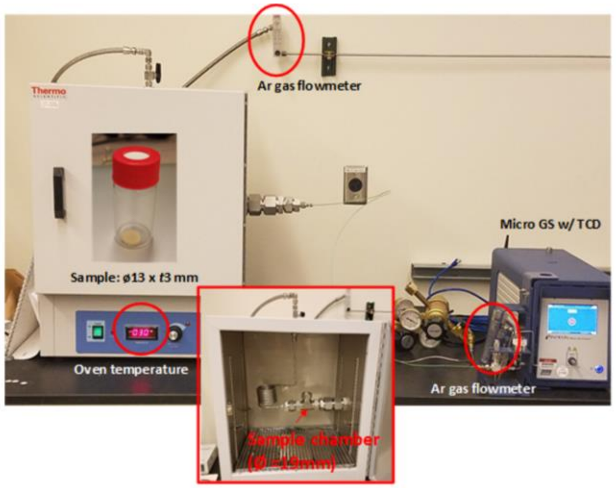
Hydrogen effects plasticizer migration through changes in solubility in rubber as shown in Helium Ion Microscopy images on left and verified by ToF-SIMs on right

Thermal Desorption Analysis (TDA) - non-equilibrium hydrogen diffusion & hydrogen saturation content

To measure non-equilibrium transport properties of hydrogen in polymer

Fick's equation at constant temperature

$$C_t = \frac{32}{\pi^2} C_0 \left[\sum_{n=0}^{\infty} \frac{exp^{\left(-(2n+1)^2 \pi^2 Dt/z^2\right)}}{(2n+1)^2} \right] \left[\sum_{n=1}^{\infty} \frac{exp^{\left(-D\beta_n^2 t/r^2\right)}}{\beta_n^2} \right]$$



sample	D1	D2	D1, Kyushu measurement*	D2, Kyushu measurement*
E1	3.199 x 10^{-10}		3.861 x 10^{-10}	
E2	4.089 x 10^{-9}			
E5	3.826 x 10^{-10}		2.239 x 10^{-10}	4.191 x 10^{-12}
E6	2.792 x 10^{-10}	2.792 x 10^{-10}		
N1	2.411 x 10^{-10}	9.824 x 10^{-11}	2.694 x 10^{-10}	3.308 x 10^{-11}
N2	2.000 x 10^{-10}	1.260 x 10^{-10}		
N5	1.908 x 10^{-10}	8.895 x 10^{-11}	3.557 x 10^{-10}	4.275 x 10^{-11}
N6	2.503 x 10^{-10}	5.641 x 10^{-11}		

To measure equilibrium transport properties of hydrogen in polymer – PNNL permeation tester

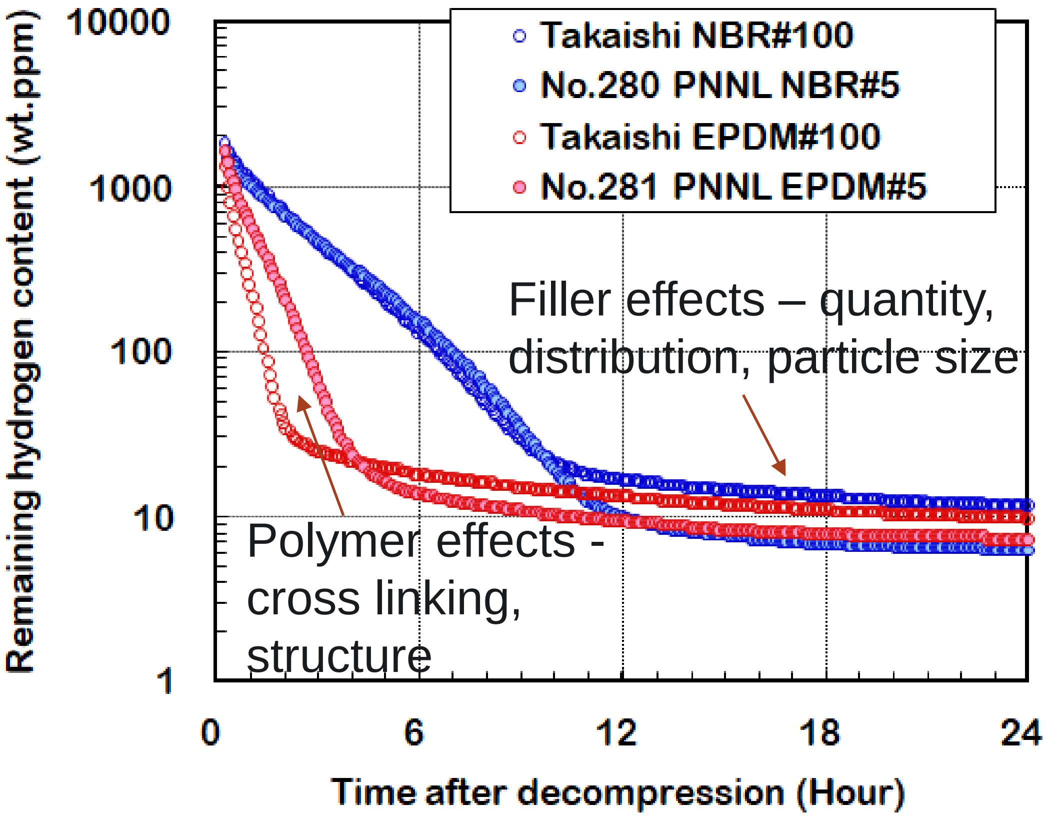
Developing database on hydrogen transport properties in polymeric materials

Progress and Accomplishments

Joint Research at Kyushu University, Japan – Understanding manufacturing process effects on materials performance against hydrogen exposure

Material performance differences between manufacturing locations in hydrogen transport properties

- The NBR exhibits a nearly equivalent elimination profile. However, it is confirmed whether there is a slight difference in the amount of CB adsorbed hydrogen after 10 hours.
- Differences in the types of carbon black (manufacturers) may have been confirmed.
- EPDM profiles differ. The amount of hydrogen found in the fitting is almost the same, but there is a possibility of chance.

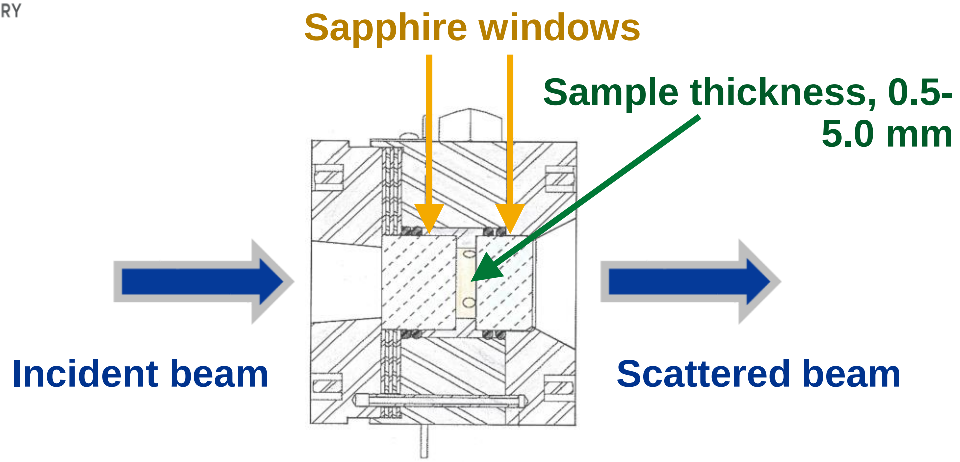


Filler effects – quantity, distribution, particle size

Polymer effects - cross linking, structure

Progress and Accomplishments

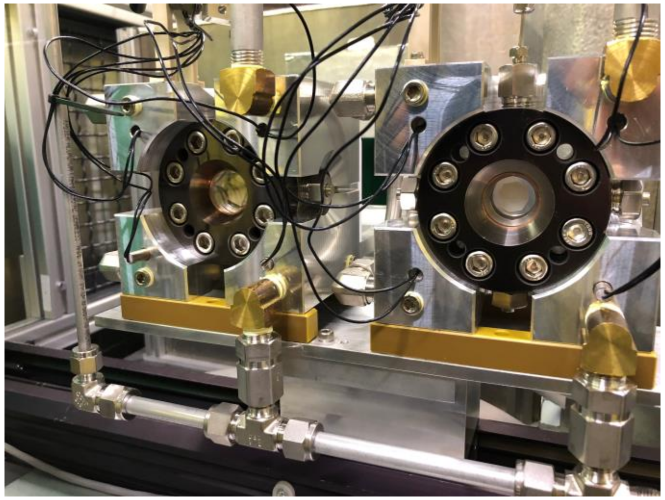
(U)SANS/SAXS shows significant differences in scattering intensities in different polymers and elastomers


Sapphire windows

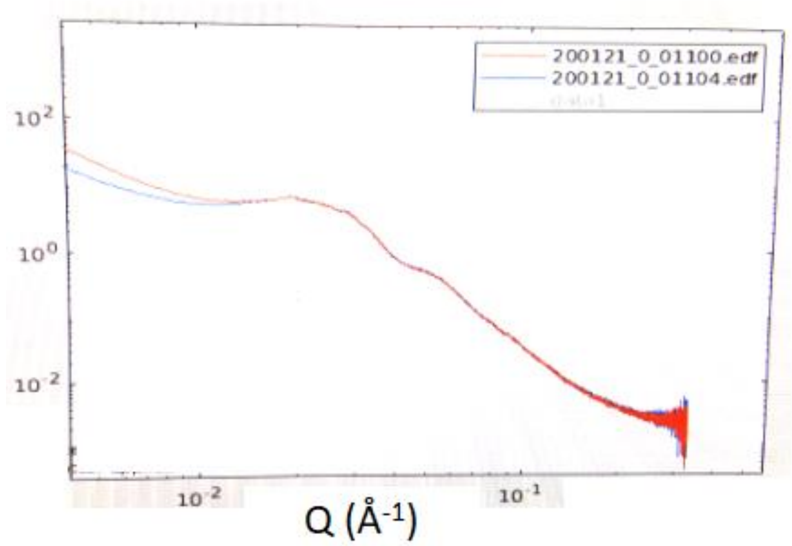
Sample thickness, 0.5-5.0 mm

Incident beam

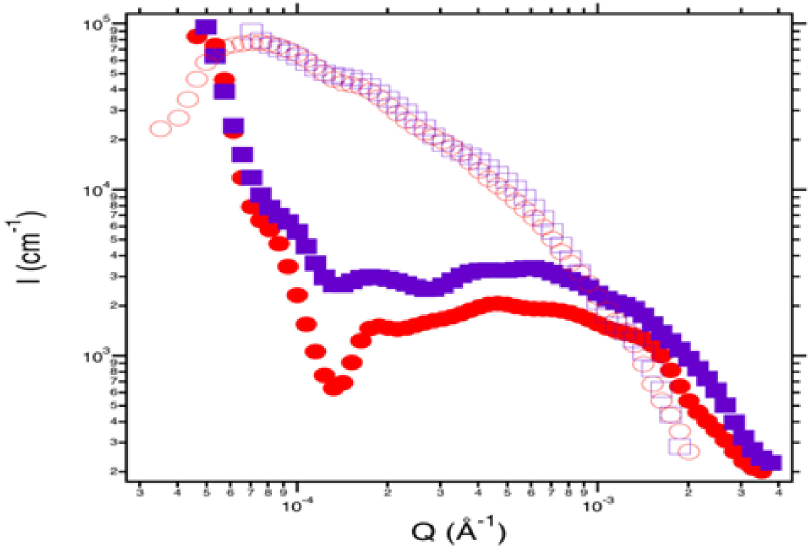
Scattered beam



- Initial (U)SANS of select thermoplastics and elastomers: PEEK, POM, HDPE, PTFE, PA-6/6, NBR, EPDM
- First P-cycled samples of thermoplastics from SNL analyzed
- Initial SAXS data for P-cycled HDPE (ORNL)
- USANS at P (four pressure cells, two staged simultaneously
  - While one sample is in beam, one is soaking (soak times were ≥ 14 hours)
- Immersion in high-pressure H_2 increases number of scatterers *for some polymers/ elastomers*



SAXS (ORNL) for HDPE Neat(blue), 100 H2 P cycles (red)
Increased large scale (small Q) porosity with cycling



USANS for PTFE (solid) and EPDM (open). H_2 saturated (purple). Neat (red). H_2 increased scatter volume in HDPE but not EPDM.

Progress and Accomplishment

H-Mat DataHub – share & dissemination

- The design for H-Mat website is complete. Development of the website has begun.
- The development and testing of the DataHUB is complete. The Ontology for projects has been created.
- DataHUB to production will roll out soon.

DOE's National Laboratories house unique, world-class capabilities that are essential to the study of hydrogen interactions with materials. These capabilities span computational modeling, imaging of hydrogen-materials interactions, and experimental evaluation of materials with applied mechanical load in high-pressure hydrogen environments. The list below provides examples of key capabilities within H-Mat. For more information, please contact h-matinfo@pnnl.gov.

[P] polymers [M] metals [S] systems

Capabilities in Mechanical Evaluation of Hydrogen Effects on Materials

Mechanical evaluation of materials in the presence of hydrogen is intended to simulate service environments and assess hydrogen-affected properties of materials. Examples of capabilities at the national labs to explore the mechanical response of materials with hydrogen include:

- Fatigue and fracture testing in high-pressure gaseous hydrogen [P] [M]
- Constant-displacement testing in high-pressure gaseous hydrogen[P] [M]
- Pressure cycling[P] [M]
- Tribology [P] [M]
- Dynamical mechanical analysis [P]
- Mechanical evaluation of materials for cryogenic hydrogen applications [S]

Capabilities in Environmental Hydrogen Interactions: Hydrogen Uptake, Transport, and Trapping

Hydrogen uptake into materials and chemical interactions with materials are the first steps of hydrogen-induced degradation phenomena. Environmental interactions are measured to inform and validate fun damental models of hydrogen transport phenomena in materials. Below are some of the unique capabilities at the national labs to probe these phenomena.

- Gas-phase permeation [P] [M]
- Thermal desorption spectroscopy [P] [M]
- Chemical analysis [P] [M]
- Hydrogen surface interactions [P] [M]

Capabilities to Evaluate Hydrogen-Microstructure Interactions

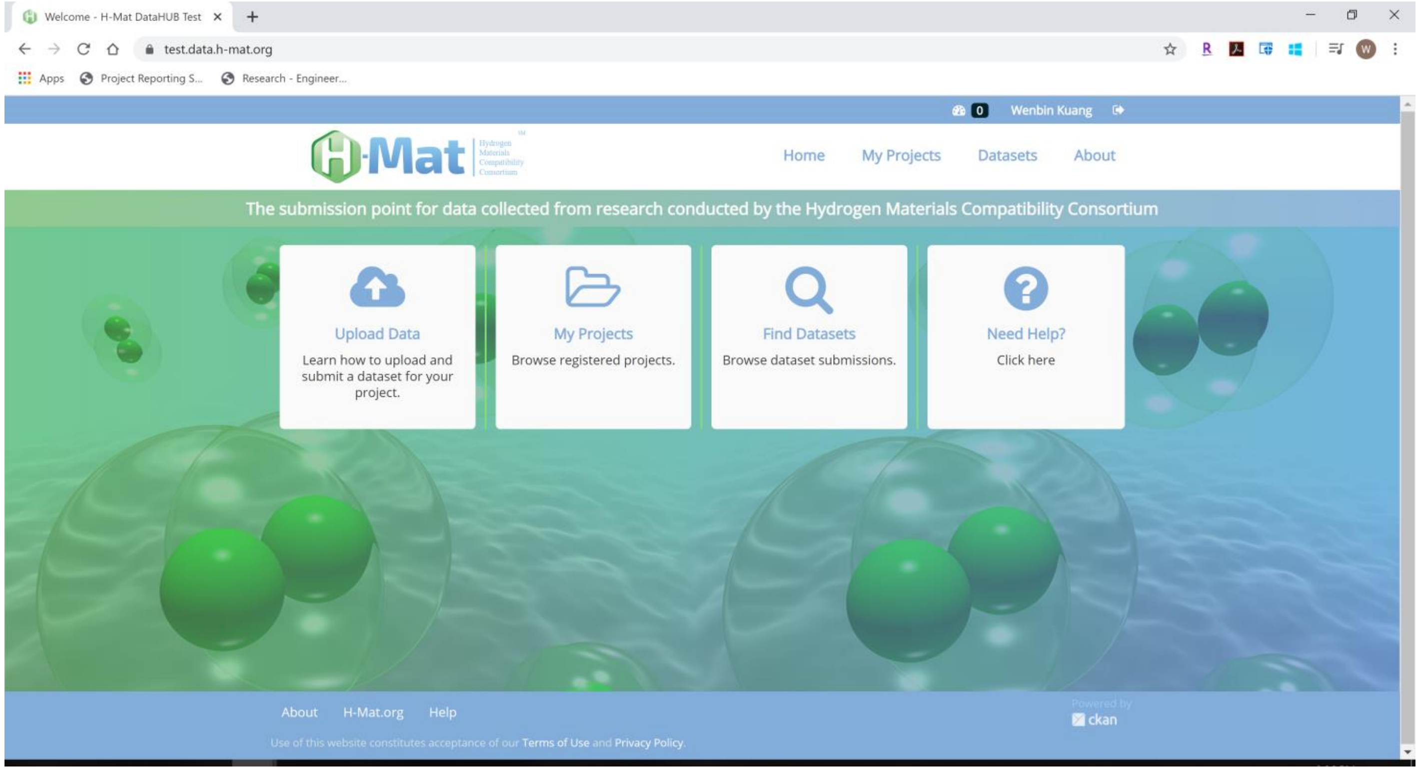
Hydrogen-materials interactions can be sensitive to the microstructure of the material. Model microstructures and simulated welds can be synthesized for mechanical and environmental interrogation. Additional tools are used to saturate (i.e., "precharge") materials with hydrogen for subsequent evaluation and measurement of relevant properties. Key capabilities at the national labs include the following.

- Thermal pre-charging [P] [M]
- Gleeble apparatus [M]
- Advanced imaging techniques [P] [M]
- Thermal Analysis [P]

Capabilities for Computational Materials Science of Hydrogen Interactions

High-performance computing and computational materials science expertise is available across the national labs for the study of hydrogen effects in materials across multiple length scales from atoms to engineering. Computational tools are available for studying materials at all relevant length scales, including (but not limited to):

- Density functional theory [P] [M]
- Molecular dynamics [P] [M]
- Dislocation dynamics [M]
- Crystal plasticity [M]
- Phase-field methods [P] [M]
- Continuum finite element methods [P] [M] [S]



The submission point for data collected from research conducted by the Hydrogen Materials Compatibility Consortium

Upload Data: Learn how to upload and submit a dataset for your project.

My Projects: Browse registered projects.

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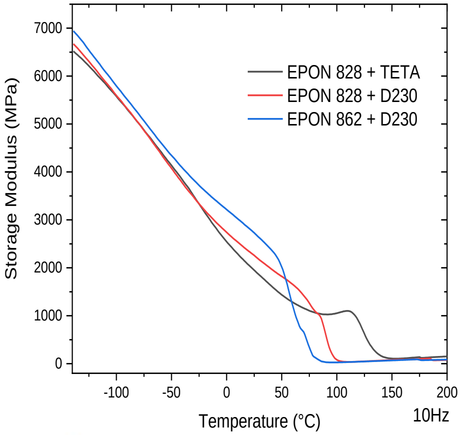
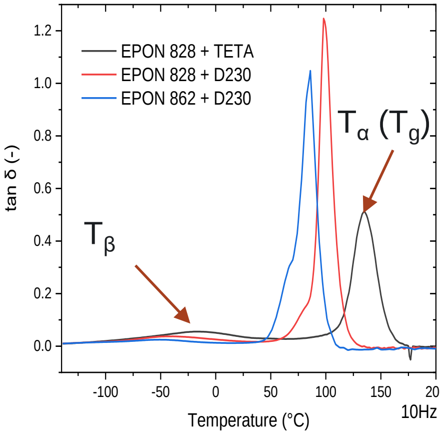
Progress and Accomplishments

Cryogenic Materials Testing and Modeling for Pressurized Storage

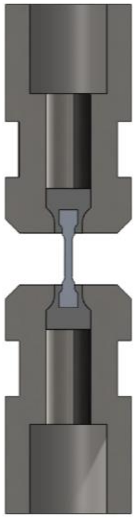
Epoxy Matrix Model Systems

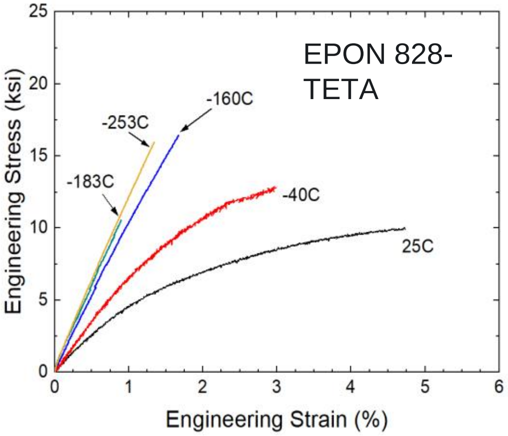


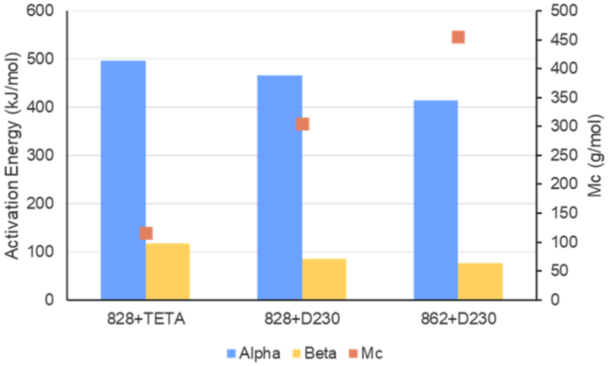


- Establish relationships between polymer structure and low temperature thermomechanical properties
  - Crosslink density
  - Thermal transitions
  - Tensile properties
- Develop relationship of H2 effects in metals properties at cryogenic temperatures

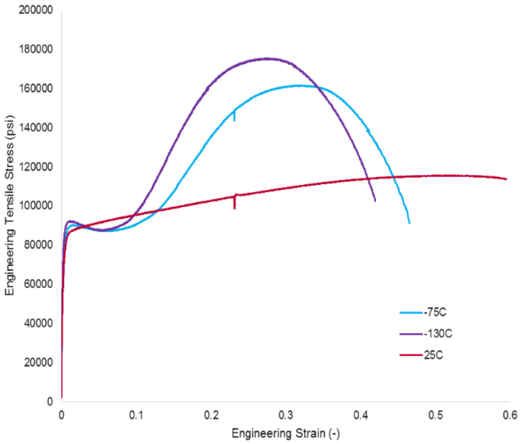
0°C to -253°C Cryogenic Materials Testing



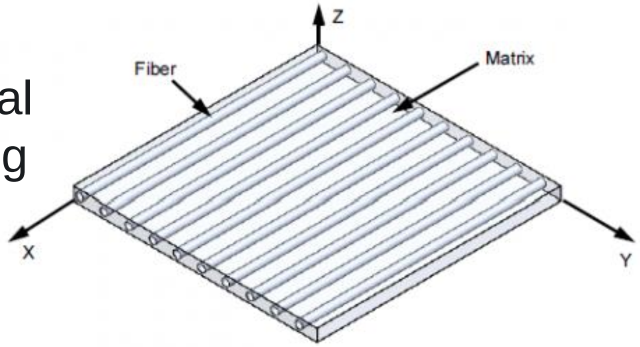




Liner Material Cryogenic Testing



Composite Material System and testing



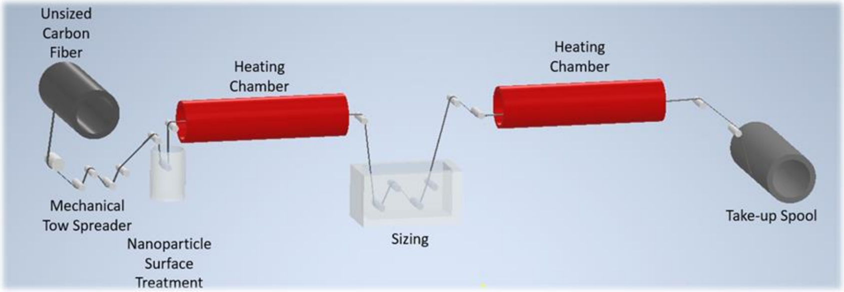
New capability and material data sets down to -253C (20K) now available for predictive modeling

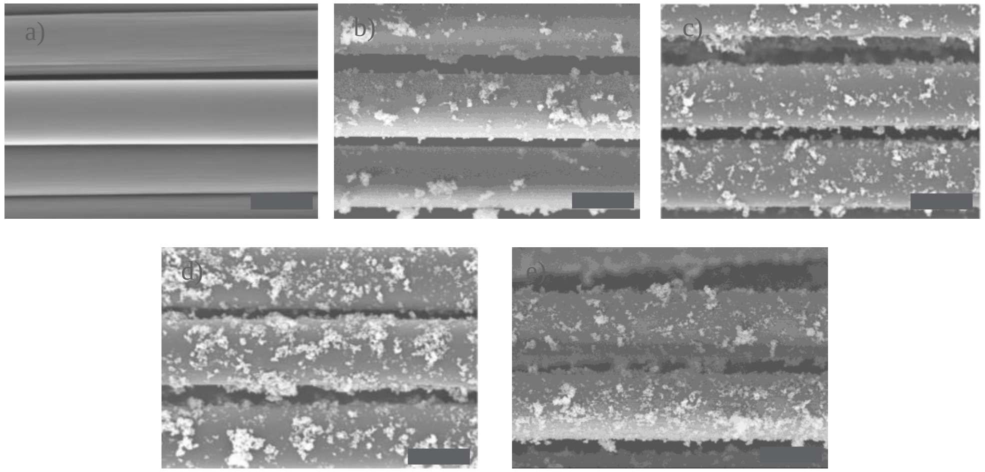
Progress and Accomplishments

Cryogenic Materials Testing and Modeling for Pressurized Storage

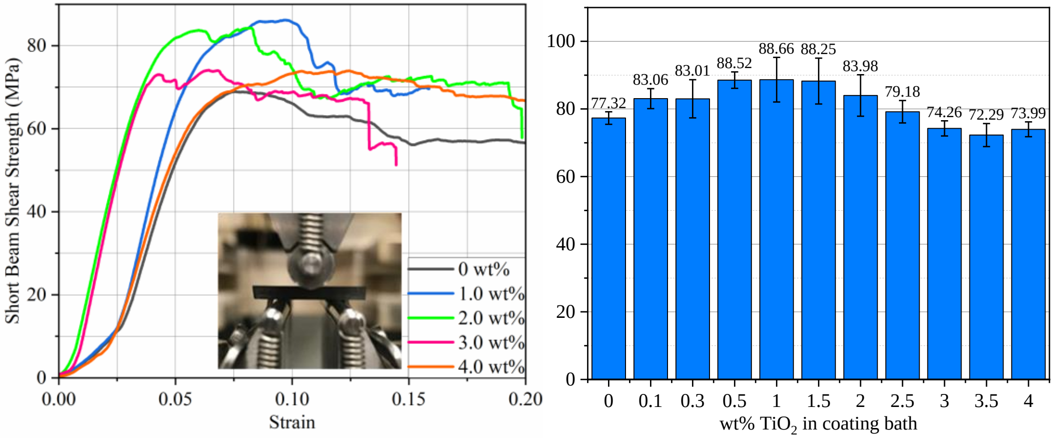
TiO_2 Nanoparticle Coating on Carbon Fiber

Continuous Feed-Through Dip Coating Process

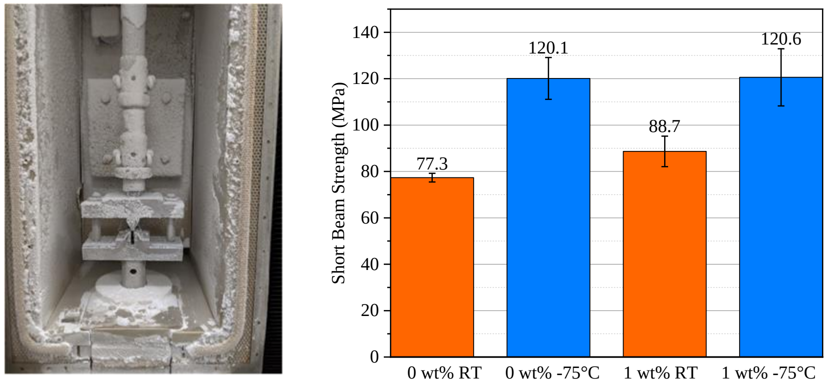




SEM images of the carbon fiber after the dip coating process using a) 0 wt%, b) 1 wt%, c) 2 wt%, d) 3 wt% and e) 4 wt% TiO_2 nanoparticle baths show good dispersion of nanoparticles on the fiber surface. Scale bars are 5µm.



The TiO_2 nanoparticles increased the interlaminar shear strength by up to 14.7% during room temperature short beam shear testing



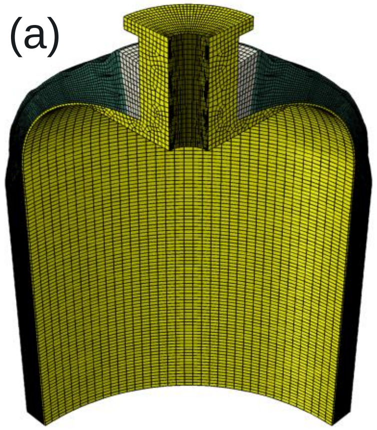
The composite strength increased at lower temperatures (-75°C), but the nanoparticles at the tested concentration did not affect the mechanical strength. So the interlaminar shear strength improvements seen at ambient temperature were not translated to low temperature tests

Goal is to increase interlaminar shear strength at cryogenic temperatures. Demonstrated >10% at ambient conditions

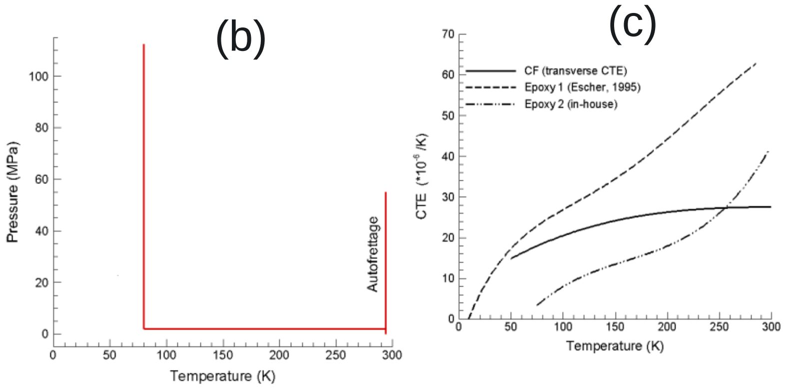
Progress and Accomplishments

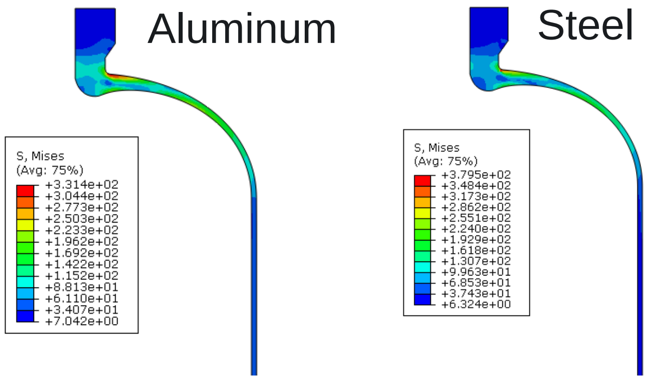
A Multiscale Modeling Approach to H2 Storage Pressure Vessels

Using multiscale modeling with experimental data for development of the constituent material behavior for the response of a cryogenic composite pressure vessel

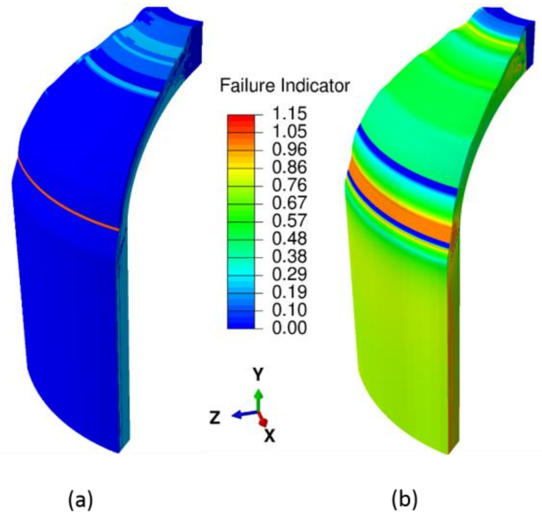


The 3D FE model for Type 3 cryo-compressed H_2 storage vessel.
(a) SS 304 liner is colored in yellow
(b) thermomechanical loading scenario applied In the FE analysis
(c) Design epoxy to reduce CTE mismatch

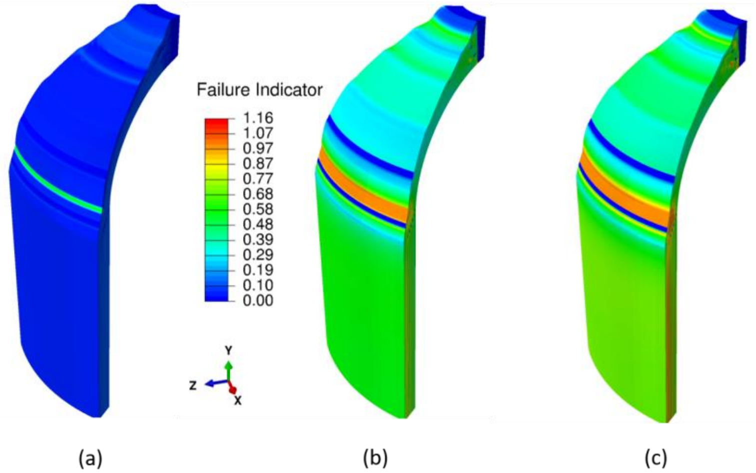




Evaluation of the aluminum and steel liner performance under pressure and at -253C



Distributions of the failure indicator values in the CF/epoxy overwrap (<u>use of Epoxy 1</u>): (a) at the end of cooling to 80 K, and (b) at 90.8 MPa burst pressure.



Distributions of the failure indicator values in the CF/epoxy overwrap (<u>use of Epoxy 2</u>): (a) at the end of cooling to 80 K, (b) at 90.8 MPa, and (c) at 104.9 MPa burst pressure.

Summary

- Polymer materials play an important role in hydrogen infrastructure components by providing both static and dynamic sealing, and high performance barriers
- H-Mat is developing a better understanding of high-pressure hydrogen gas interaction with polymers for the hydrogen community that will increase reliability and durability of the hydrogen infrastructure
- Developing multiscale modeling (atomistic, meso scale, component scale) efforts to build predictive tools for understanding polymer-hydrogen interaction and damage mechanism
- Initial studies using *in-situ* DMA demonstrated pseudo-plasticization effects on polymer mechanical properties
- Helium ion microscopy used to reveal that phase separation of plasticizer and formation of large voids (30-40 um) are induced by hydrogen
- Comparison of He and H2 gas experiments show hydrogen effects in polymers are increased over He at the same pressure levels
- Comparison between virgin sample, 27.6 MPa hydrogen exposed, 90 MPa hydrogen exposed, and 90 MPa hydrogen exposed for 12 cycles, showed profound morphological changes created by pressure cycling in high pressure hydrogen
- TDA analysis studied the effects of fillers and plasticizer on transport properties of model materials – plasticizer increasing hydrogen permeability while filler decreasing it

Acknowledgements

This work was fully supported by the U.S. Department of Energy (USDOE), Office of Energy Efficiency and Renewable Energy (EERE), Fuel Cell Technologies Office (FCTO) under Contract Number DE-AC05-76RL01830. We also acknowledge support from the Environmental Molecular Sciences Laboratory (EMSL). EMSL is a national scientific user facility sponsored by the Department of Energy's Office of Science, Biological and Environmental Research program that is located at Pacific Northwest National Laboratory.

DISCLAIMER

Response to previous year's reviewers' comments

- H-Mat is a new project and was not reviewed last year

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Thank you