

- Two alternative fuel (hydrogen) R&D projects funded by DOT/PHMSA and coordinated by Louis E. Hayden, P.E.
- Projects are constructed, executed and coordinated to provide technical input to the pipeline industry, SDO's (Standards Development Organizations) and regulators (DOT/PHMSA)
- Technical data developed by these projects will be useful to pipeline designers, operators and owners. Through the use of project results, pipeline design, construction and operation costs can be reduced.

- All pipeline steel and weld samples tested have complete chemistries and actual tensile and yield strengths documented. Each sample has its microstructure evaluated with constituent % documented.
- Both project groups are utilizing many of the same donor steels in their tests so that results can be compared and evaluated.
- A wide array of old, new and just installed pipeline steels are included in the scope of both projects.

# Accomplishment & Capability

## Base materials and welds :ORNL/ UT

### Accomplishments

- The baseline fatigue-crack-growth-rate experiments (in air ) were finished on two kinds of base pipeline steels [Alloy B (Fe-0.05C-1.52Mn-0.12Si-0.092Nb, weight percent (wt.%) and alloy C (Fe-0.04C-1.61Mn-0.14Si-0.096Nb, wt.%) at different frequencies (10 Hz, 1 Hz, and 0.1 Hz) and different R ratios (0.1 and 0.5).
- The influence of frequency and R ratio on fatigue crack growth rate were compared.
- A neutron experiment on studying the deformation behavior of the crack tip of X 52 and X 70 pipeline steels was completed ( in air).

### Capabilities

- The facilities at ORNL enable us to perform cyclic fatigue tests on the weld material of an X-70 pipeline steel in hydrogen atmospheres (up to 33 MPa) under different loads, with a frequency of 2 Hz.
- Fatigue-crack-growth behavior of an X-70 pipeline weld will be investigated, and the deformation behavior at the fatigue-crack tip will be studied by in-situ neutron and synchrotron diffraction.
- Develop computational tools to model the fatigue process of pipeline steel welds.

### Experimental Facilities for Materials Testing in High Pressure Hydrogen Available at Oak Ridge National Laboratory (ORNL)



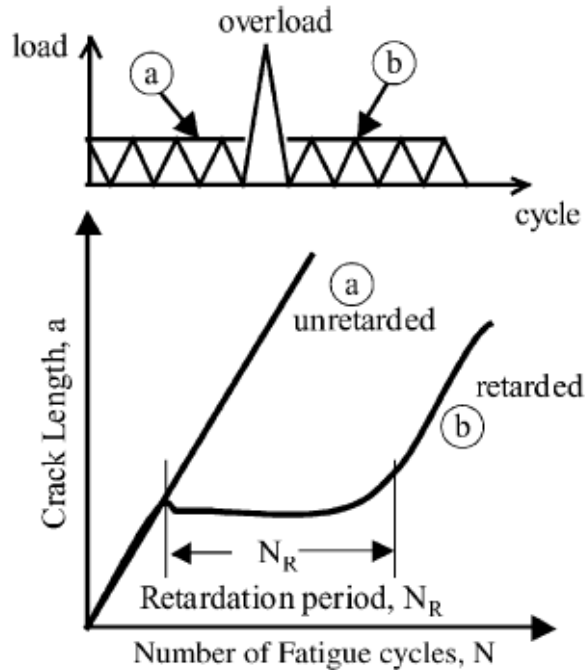
#### Key features

- Room temperature gas pressures up to 33 MPa (4,800 psi)
- Tensile Strain rates down to 1E-6/sec
- Test loads up to 18 kN (4,000 lbs)
- Flexible specimen geometry
- Computer-controlled valves and data acquisition
- Fracture and fatigue testing capabilities

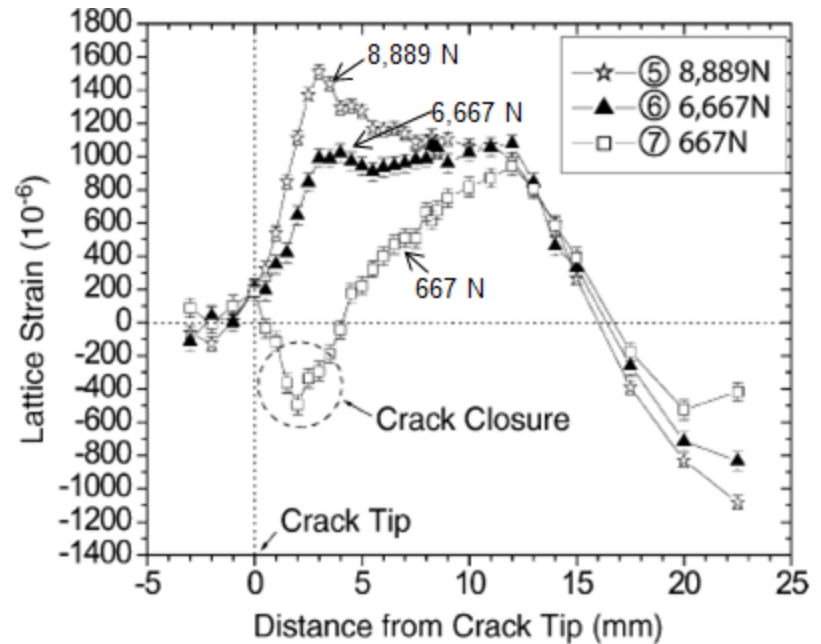
### Spallation Neutron Source (\$1.4 B) Site ORNL, Oak Ridge, TN



# An Example of Neutron Study



The effect of overload on fatigue-crack-propagation



In-plane lattice-strain profiles in front of the crack-tip at applied loads of 8,889 N, 6,667 N and 667 N.

- Neutron experiment was performed to help understand the effect of overload on the fatigue life of 316 LN stainless steel.
- At the tensile load of 667 N following the overload to 8,889 N, compressive lattice strains of up to about  $-600 \mu\epsilon$  were observed within 4 mm from the crack-tip, indicating the plasticity-induced crack-closure. This could subsequently decrease the crack-growth rate, and increase the fatigue life.

S.Y. Lee, P.K. Liaw, H. Choo, R.B. Rogge, Acta Materialia, 2011,59: 485-494.

S.Y. Lee, H. Choo, P.K. Liaw, K. An, C.R. Hubbard, Acta Materialia, 2011,59: 495-502.

Y. Sun, H. Choo, P.K. Liaw, Y. Lu, B. Yang, D.W. Brown, M.A.M. Bourke, Scripta Materialia, 2005, 53: 971-975.

## Proposed Work

In-situ neutron/synchrotron experiments for pipeline-steel welds in hydrogen atmosphere.

## Purposes

- Gain a fundamental understanding of the fatigue-deformation behavior of pipeline steel welds.
  - Since a plastic zone generally produces around the fatigue-crack tip, the crack needs to pass through this plastic zone. Thus, fatigue-crack-growth behavior is controlled by deformation zones that exist around the crack tip.
  - Using neutron/synchrotron diffraction, we will be able to collect the strain information around the crack tip, thus probing fatigue mechanisms.
- Study the hydrogen effect on the fatigue behavior of pipeline-steel welds.
  - The in-situ neutron/synchrotron experiment will be very close to the real situation (pipeline steel welds in service).
  - By running the test in air and hydrogen atmosphere, we will be able to detect the influence of hydrogen on the fatigue process of pipeline steel welds.
- Develop computational tools to model the fatigue process of pipeline steel welds.
  - The developed models quantify the lattice-strain behavior, which can be compared with neutron/synchrotron results.

# Future Plan (Cont'd)

- The neutron/synchrotron results can provide significant information to help improve the model, which can be used to better predict the fatigue life of pipelines to be used for the transportation of hydrogen.

## Technical Approach

- To create a safe hydrogen environment within the neutron/synchrotron, we need to make a small chamber filled with a limited amount of hydrogen.
- The test chamber needs to be thin enough, so that it will not block the neutron/synchrotron beam. Specimen loading will be accomplished using Micro-Electro-Mechanical (MEMS) Technology.
- Due to the combination of size, hydrogen, and neutron/synchrotron, we will need to build a special fatigue test system.

## Impacts

- The developed in-situ neutron/synchrotron study of crack-tip deformation behavior will greatly help the production of better pipelines for the transportation of hydrogen and other gasses, and assure the structural integrity of pipeline systems.
- Because of the small-scale sample, chamber, and special test system, we may create a new standard of in-situ neutron/synchrotron mechanical testing in hydrogen.

## Understanding and mitigating hydrogen embrittlement (HE) in high-strength pipeline steel welds

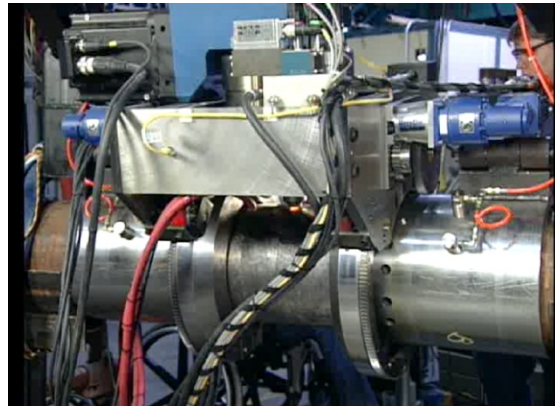
- Establishing weld microstructure-property relationship for HE:
  - *Characterization of weld microstructure inhomogeneity*
  - *In-situ testing of weld mechanical properties in high-pressure hydrogen*
- Improving weld resistance to HE:
  - *Solid-state friction stir welding*
  - *HE-resistance filler metal chemistry*

***\* Developed tools and methods can be extended to other welding-related issues in pipelines such as stress-corrosion cracking.***

# Capability: Automated Field Welding Technology

Solid-state friction stir welding (FSW) of steel pipelines

- “Forge” welding with material flow / recrystallization driven by a rotating tool pin



*Prototype FSW equipment for field welding of steel pipelines*

*Superior tensile and toughness properties*



*Projects sponsored by U.S. Department of Energy (DOE) Advanced Manufacturing and Fuel Cell Technologies Programs*

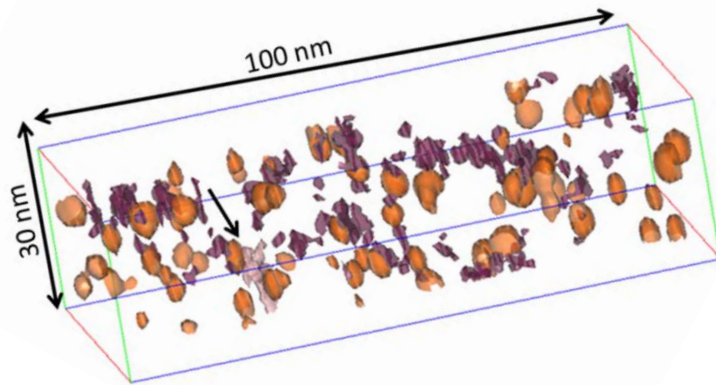
- Scalable to thicker steel section through multi-layer, multi-pass FSW



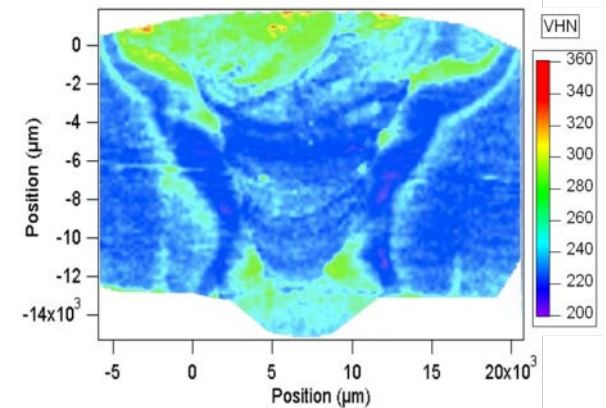
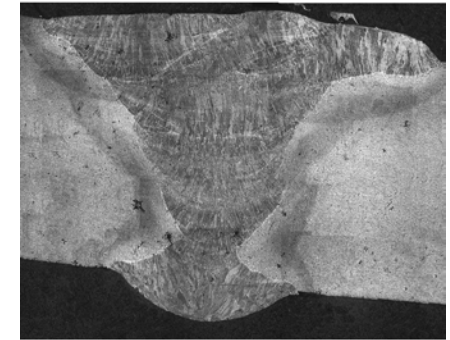


## Specially-tailored filler metal:

- Microstructure characterization and modeling tools: (e.g., micro-hardness, atom-probe tomography, and computational thermodynamics and kinetics)
- Increasing the amount of retained austenite for improving resistance to hydrogen embrittlement



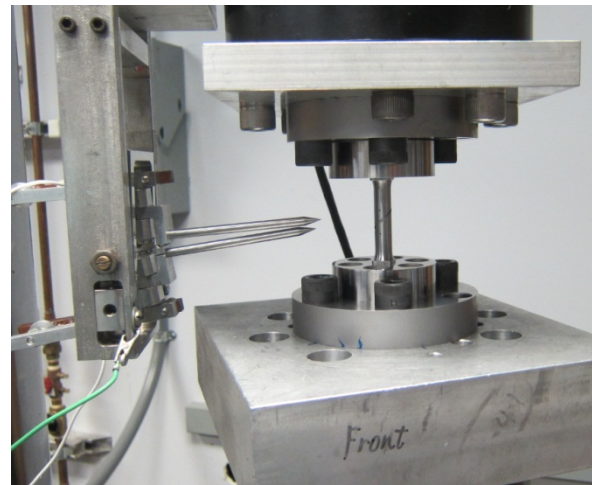
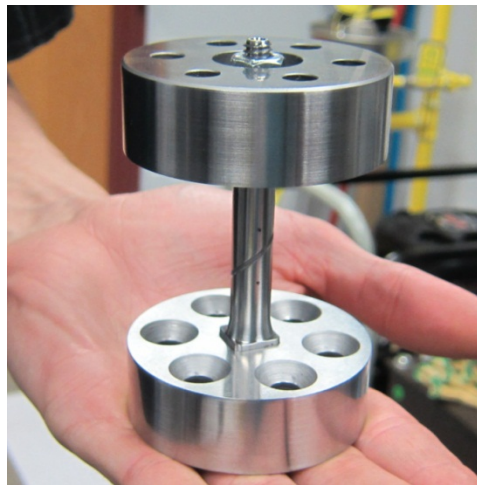
Advanced microscope resolving distribution of precipitates for studying steel precipitate hardening mechanism



*Highly non-uniform microstructure in an X-80 weld revealed by micro-hardness mapping*

# Capability: Mechanical Testing of Weld Properties: ORNL

- Cost-effective mechanical testing methods tailored for inhomogeneous microstructure



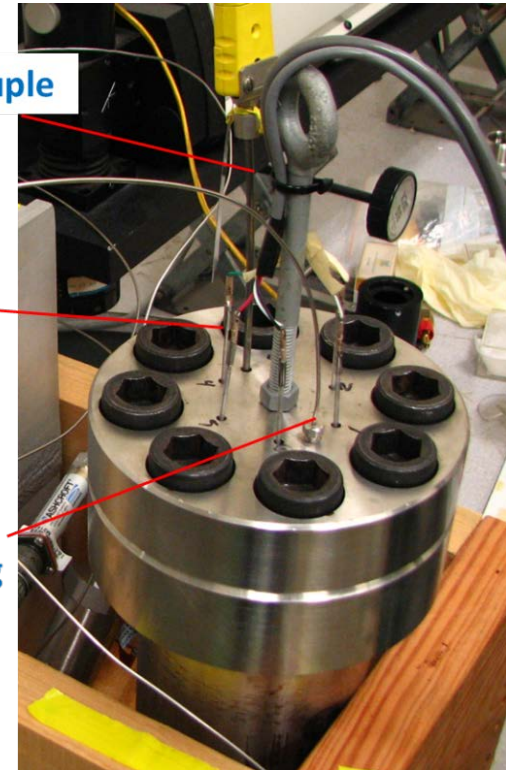
*Testing setup in ambient air*

Special testing method using spiral notch torsion test (SNTT) for studying hydrogen embrittlement of pipeline steels and welds

Thermo-couple

Wires for strain gages

H<sub>2</sub> port for charging / discharging



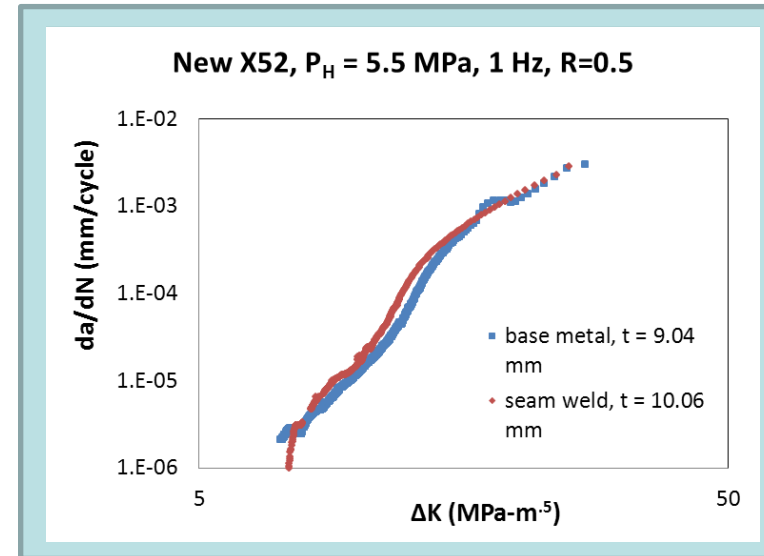
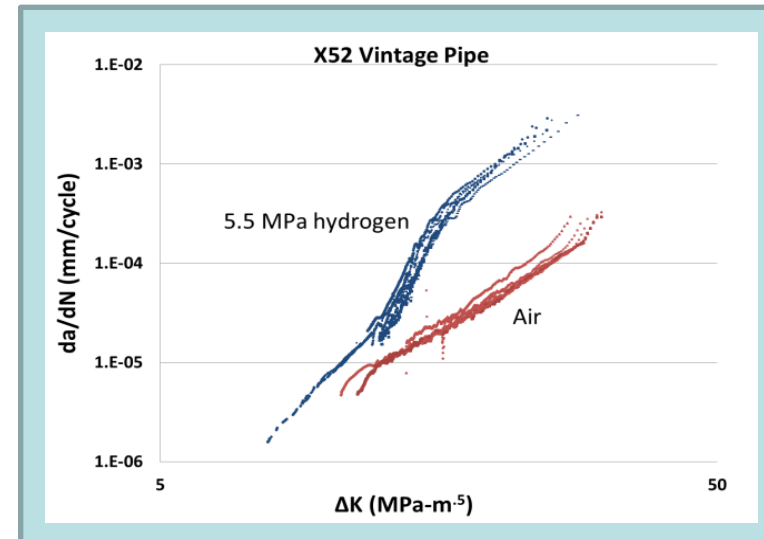
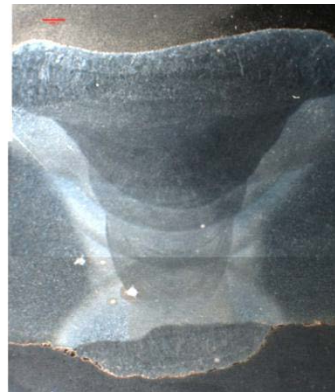
*In-situ testing setup in high-pressure hydrogen*

- **Friction stir welding:**
  - *FSW tool life and joining of thick section*
  - *Mechanical properties of FSW joint and codification*
- **Welding filler metal:**
  - *Balance of tensile strength and fracture toughness*
  - *Weldability and defects*
  - *Filler metal cost*
- **Microstructure-processing-properties database for welds**

# Accomplishments NIST/CRES

- Generating fatigue data on pipeline steels in pressurized hydrogen gas for codes and standards to increase safety and reliability, while allowing more economical designs
- Developed a unique method for rapid generation of fatigue data (<10% time) that is highly precise (low measurement uncertainty)
- Conducting preliminary studies on the behavior of seam and girth welds
- Generating a model to predict lifetimes in a hydrogen (or other) gas environment

Materials Conditions	X52 Vintage	X52 New	X70 A	X70 B
Air, 1 Hz	5 of 5	5 of 5	5 of 5	5 of 5
5.5 MPa, 1 Hz	5 of 5	5 of 5	0 of 5	0 of 5
34.5 MPa, 1 Hz	5 of 5	5 of 5	5 of 5	5 of 5
5.5 MPa, 0.1 Hz	3 of 3	2 of 2	3 of 3	2 of 2
34.5 MPa, 0.1 Hz	5 of 5	5 of 5	0 of 5	0 of 5
5.5 MPa, 0.01 Hz	0 of 1	0 of 1	0 of 1	0 of 1
34.5 MPa, 0.01 Hz	0 of 1	0 of 1	0 of 1	0 of 1



Mechanical testing in pressurized gas environments,  
 $P_H \leq 140$  MPa, including hydrogen and other corrosive  
gases

Dynamic tests,  $f = 0.01 - 1$  Hz

Static tests: tensile and fracture

Servo-hydraulic load frames: 250 kN capacity and 100  
kN

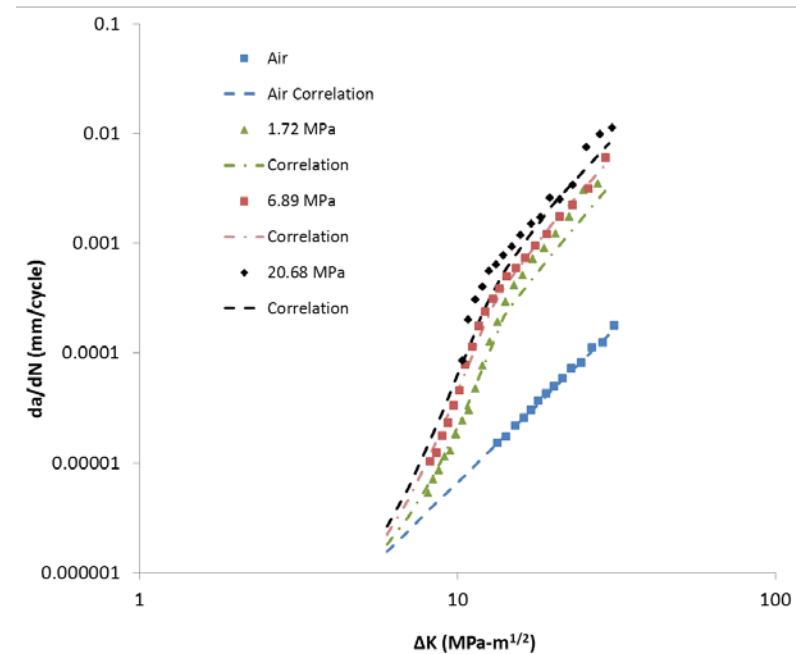
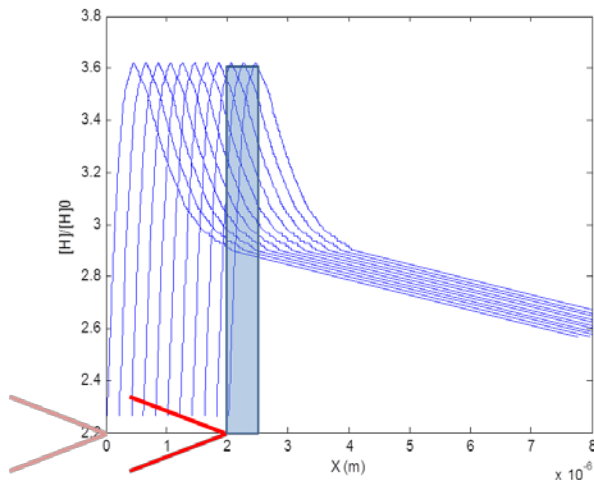
Test chambers: 140 MPa single specimen, and 34  
MPa multi-specimen

Multi-specimen fatigue tests using CT specimens  
(according to ASTM E647)

Fractography and microstructural characterization  
Hydrogen pre-charging at 14 MPa and 450 °C



- Phenomenological modeling
- Constitutive modeling
- Hydrogen diffusion coupled with FCG modeling
- Pipeline lifetime modeling



## Welds and HAZ

- Systematic study of fatigue properties of welds and HAZs in corrosive gas environments
- NIST can measure fracture using C(T)s as a baseline for ORNLs measurements using the torsion specimen
  - Both sites can measure the same materials to establish the torsion method
- Explore using SE(T) specimens to measure fracture in a corrosive gas environment. Currently used by the pipeline industry to:
  - more closely approximate the constraint conditions in service
  - provide data for strain-based design

## Hydrogen Induced Corrosion (Embrittlement)

- Effect of filled traps
  - On fatigue properties
  - Investigate methods to identify high- and low-energy trapping sites
  - If successful, investigate methods to verify whether or not hydrogen remains in high-energy sites while stressed

## Modeling

- Expand the model to other corrosive environments and pertinent grades of steel, providing industry with outputs of interest.
- Produce experimental data to support the development of a model that provides industry with cycles to initiation and lifetimes for the above conditions.