Sensor advanced manufacturing and structural materials characterization

Advanced Sensors and Instrumentation Annual Webinar

October 29, November 5, November 12 , 2020

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Project Overview

- Goal and Objective
  - To demonstrate and benchmark the performance reliability of advanced manufactured melt wire materials when compared to standard melt wires with zinc, aluminum and tin.
  - Creep behavior is critical for safety concerns and longevity of current and future nuclear reactors. In-situ creep testing capability offers more control over experimental variables, provides more details regarding creep behavior, and gives you improved accuracy compared to non-instrumented tests.
- Participants
  - Kiyo Fujimoto, Kunal Mondal, Lance Hone, Malwina Wilding, Kurt Davis, Richard Skifton, Eric Larsen, Ashley Lambson, Michael McMurtrey
- Schedule
  - Complete laser-based non-destructive adhesion strength measurements for advanced manufactured sensor adhesion characterization (7/23/21)
  - Characterize bi-metallic ink for use in melt wires to expand passive peak temperature resolution (7/23/21)
  - Develop of real-time, in-pile test rigs for characterization of nuclear components structural materials (9/30/21)

Summary of accomplishments

- FY20 Milestones
  - Demonstration of non-visual analysis of miniaturized encapsulated melt wire arrays for the detection of peak irradiation temperature in reactor core (2/21/20)
  - Fabricate and characterize miniaturized encapsulated melt wire arrays for the detection of peak irradiation temperature in reactor core (9/30/20)
  - Complete database of available ink materials for sensors fabrication (9/30/20)
  - Complete selection of materials for LVDT based creep test rig demonstration (1/24/20)
  - Perform out of pile test of LVDT based creep test rig at PWR prototypical conditions (7/10/20)

Summary of accomplishments

- Synthesis of molybdenum nanoparticles from wet chemical methods. Requires optimization.
- Demonstrated the ability of synthesizing bi-metallic nanoparticles of Bi/Pt from wet chemical methods.
- Completed comparison between standard and advanced-manufactured melt wires of tin, aluminum and zinc.
  - Established an evaluation method for assessing the performance of advanced manufactured melt wires.
- Performed preliminary measurements of vibrational resonances of thin films deposited on substrates using direct-write techniques.
  - Goal is to utilize laser ultrasonics for non-destructive characterization of film/substrate adhesion in sensors fabricated using advanced manufacturing.
- The phase-field model was further developed to better model sintering physics.
  - The model was parameterized and run Pt, Mo and Nb on alumina.
- Tested Creep Test Rig with SS 304 sample at 2500 psi and 300 ℃ in autoclave and the NI DAQ system for continuous recoding of measurements and comparison to Halden DAQ system.
- Supporting in-pile creep testing rigs for the Navy tests that will be conducted at ATR.

Technology Impact

Describe how this technology:

- *Advances the state of the art for nuclear application*

Utilizing advanced manufacturing facilitates the production of novel sensor designs for in-pile sensors and instrumentation designs that are not otherwise achievable through classical fabrication techniques. With the closure of the Halden Reactor, this work maintains and develops mechanical testing (in-situ creep and stress-relaxation) capabilities that would otherwise be lost.

- *Supports the DOE-NE research mission*

Facilitates the development of advanced sensors and instrumentation with cross-cutting technology development. In-situ creep testing capability is currently being designed into future Navy tests that will be conducted at ATR; as well as, in-situ stress relaxation capability part of the FIDES JEEP proposals for upcoming FY21 timeframe.

- *Impacts the nuclear industry*

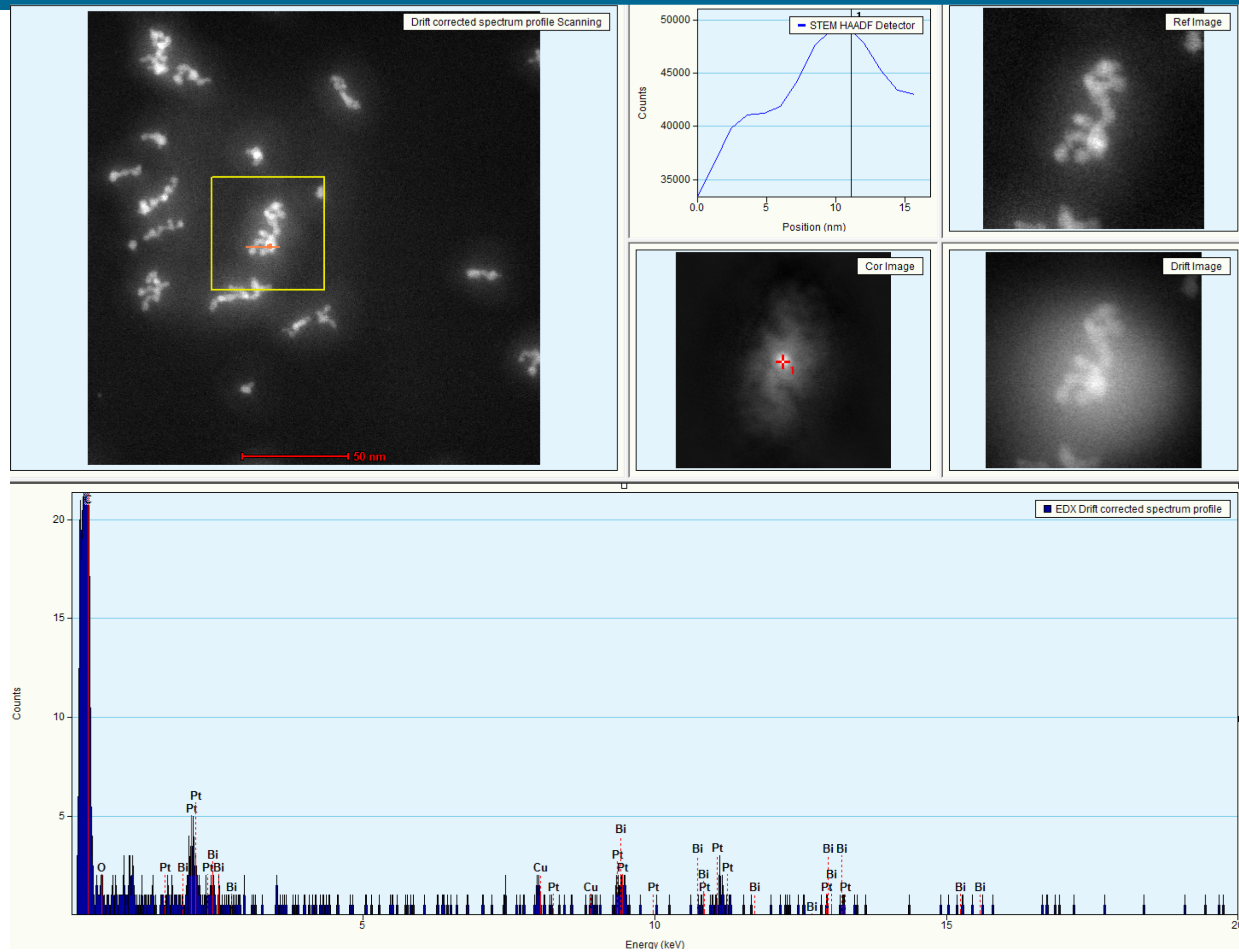
Development of advanced methods and capabilities to enable transformative sensor technology for in-pile monitoring and in-situ analysis of fuels and materials. Real-time creep and stress-relaxation measurements are a critical asset in the design of new materials for fuel, cladding, and structures in the next generation as well as existing nuclear reactors.

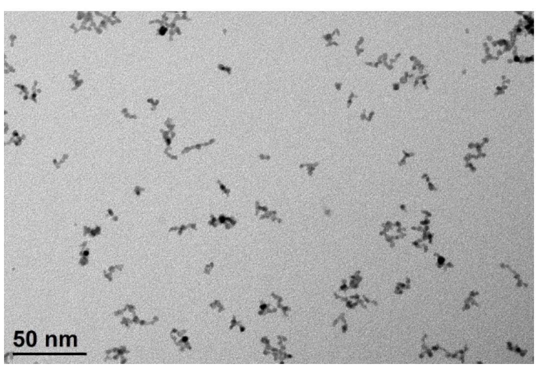
- *Will be commercialized*

Interest from industry has already been received for AM melt wires (Westinghouse). However, still in early research and development stages.

Accomplishments: Ink development

- Bismuth-Platinum nanoparticles
- TEM –EDS confirmed alloyed nanoparticles
- Continued development in to new ink materials





Nickel	Black Phosphorus	h-BN	Carbon nanotubes	Tellurium inks	Niobium	Tungsten	Tin
ZnO	MXene (Ti3C2)	Graphene Oxide	Bismuth Selenium Telluride	Black Phosphorus	Molybdenum	Zinc	Aluminum
Bismuth Telluride (2D)	MoS2	Polyamide	Bismuth antimony telluride	Platinum	Cobalt	Indium	Cobalt Oxide (Co2O3)
Graphene (2D)	WS2	Polyaniline	Antimony Telluride	Titanium	Iron	Alumina (Al2O3)	

Accomplishment: Process control modeling

A three-phase grand-potential-based phase-field model is used to predict ideal sintering conditions for nano-particles inks.

Properly capturing diffusivity is essential to model sintering. This year, an anisotropic diffusivity tensor was implemented in order to model fast diffusion along grain boundaries and surfaces. The novel three-phase mathematical model for the diffusivity is shown on the right.

$$\mathbf{D} = \mathbf{D}^{\alpha}_{blk} + \mathbf{D}^{\beta}_{blk} + \mathbf{D}^{\zeta}_{blk} + \mathbf{D}^{\alpha-\alpha}_{bnd} + \mathbf{D}^{\alpha-\beta}_{bnd} + \mathbf{D}^{\alpha-\zeta}_{bnd} + \mathbf{D}^{\beta-\zeta}_{bnd}$$

$$\mathbf{D}^{\alpha}_{blk} = \mathbf{I}\, D^{\alpha}_{v} \sum_{i=1}^{n^{\alpha}} \alpha_i, \qquad \mathbf{D}^{\beta}_{blk} = \mathbf{I}\, \beta\, D^{\beta}_{v}\,, \qquad \mathbf{D}^{\zeta}_{blk} = \mathbf{I}\, \zeta\, D^{\zeta}_{v}$$

$$\mathbf{D}^{\alpha-\beta}_{bnd} = D^{\alpha-\beta}_{bnd} \sum_{i=1}^{n^{\alpha}} \alpha_i \beta\, \mathbf{T}^{\alpha-\beta}_{ij} \qquad \mathbf{T}^{\alpha-\alpha}_{ij} = \mathbf{I} - \frac{\nabla\alpha_i - \nabla\alpha_j}{|\nabla\alpha_i - \nabla\alpha_j|} \otimes \frac{\nabla\alpha_i - \nabla\alpha_j}{|\nabla\alpha_i - \nabla\alpha_j|}$$

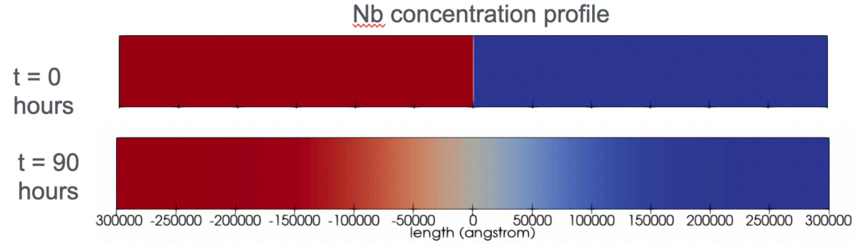
$$\mathbf{D}^{\alpha-\alpha}_{bnd} = D^{\alpha-\alpha}_{bnd} \sum_{i=1}^{n^{\alpha}} \sum_{j\neq i}^{n^{\alpha}} \alpha_i \alpha_j \mathbf{T}^{\alpha-\alpha}_{ij} \qquad \mathbf{T}^{\zeta} = \mathbf{I} - \frac{\nabla\zeta}{|\nabla\zeta|} \otimes \frac{\nabla\zeta}{|\nabla\zeta|}$$

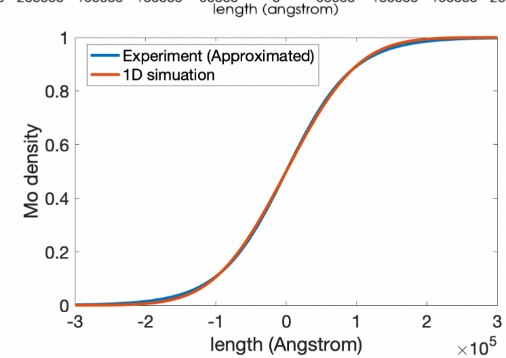
$$\mathbf{D}^{\alpha-\zeta}_{bnd} = D^{\alpha-\zeta}_{bnd}\, \zeta^2 (1-\zeta)^2\, \mathbf{T}^{\zeta} \qquad \mathbf{T}^{\alpha-\beta}_{i} = \mathbf{I} - \frac{\nabla\alpha_i - \nabla\beta}{|\nabla\alpha_i - \nabla\beta|} \otimes \frac{\nabla\alpha_i - \nabla\beta}{|\nabla\alpha_i - \nabla\beta|}$$

$$\mathbf{D}^{\beta-\zeta}_{bnd} = D^{\beta-\zeta}_{bnd}\, \zeta^2 (1-\zeta)^2\, \mathbf{T}^{\zeta}$$

Significant effort was spent on parameterization and verification in FY20.

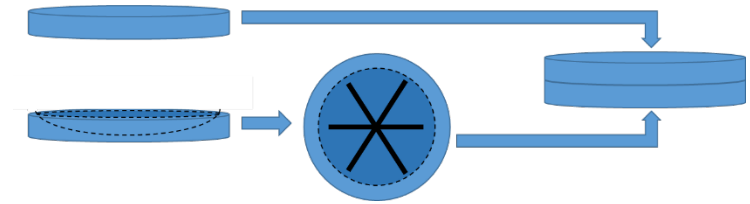
- Verification of the model was carried out through studies on geometric equilibrium as well as one-dimensional diffusivity behavior (shown on right).
- Parameterization for multiple systems was conducted through a combination of literature review and ab initio studies performed at Boise State.

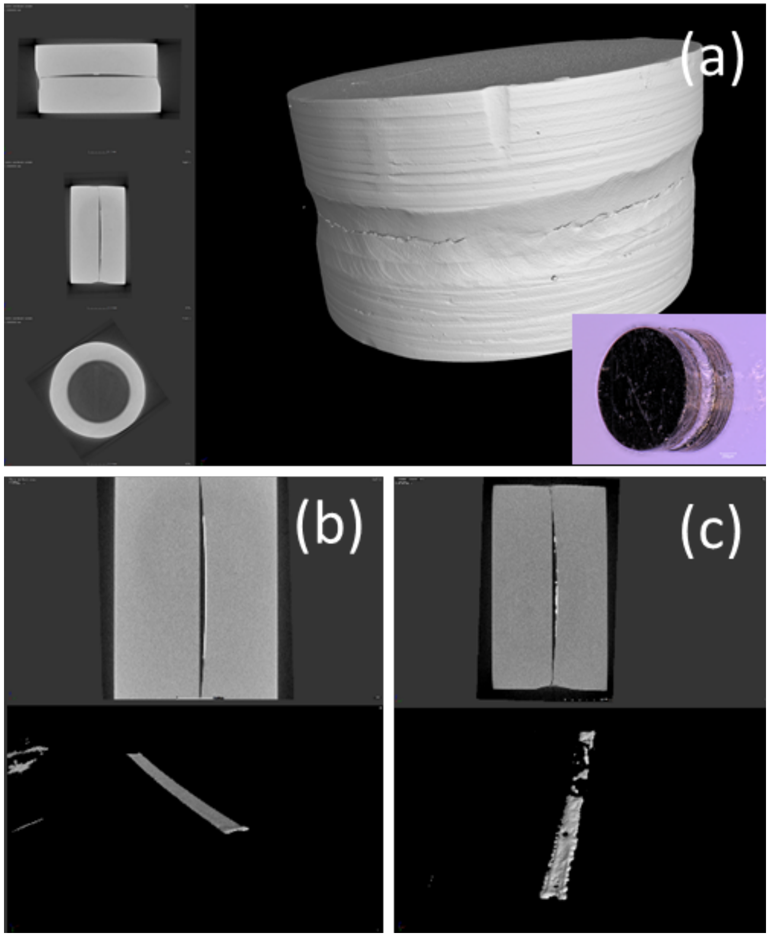


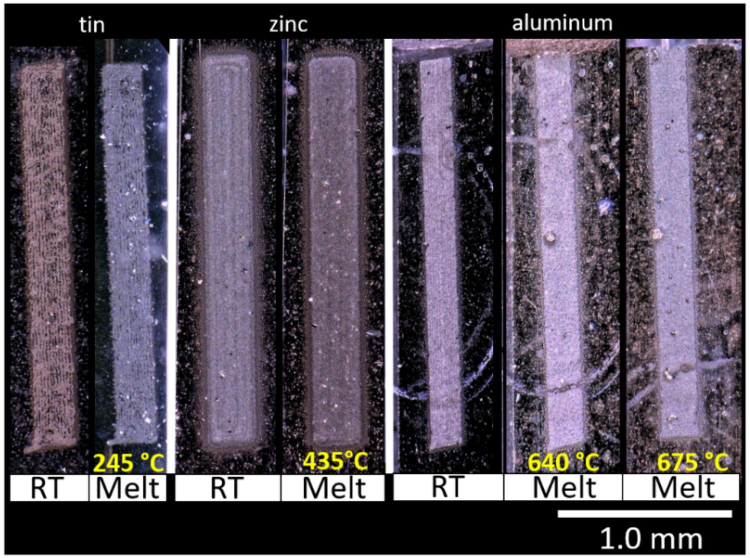


Accomplishments: Melt wire chips

- Successfully demonstrated X-ray computed tomography as a tool to “read” sealed melt wire chips
- Benchmarked printed melt wires against traditionally manufactured melt wires
  - Through differential scanning calorimetry and furnace testing it was determined that the performance of the advanced manufactured melt wire capsule was consistent with that of the standard melt wire capsule

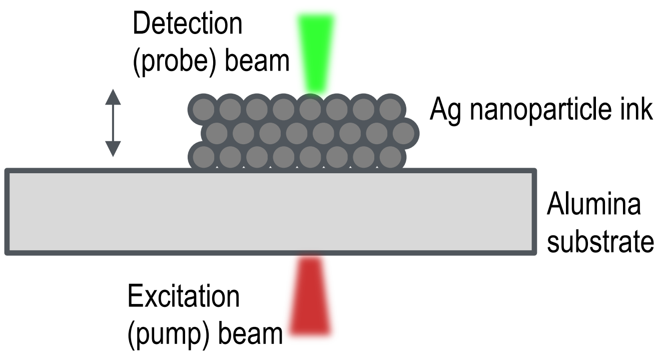




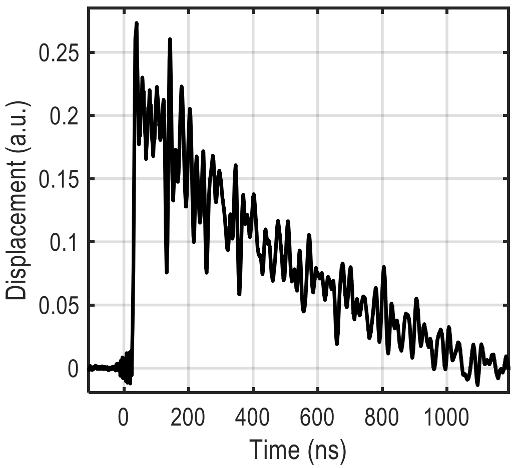


Accomplishments: Laser-based adhesion measurements

- Utilized a non-contact pump-probe laser ultrasonic technique for preliminary measurements fixed-free vibrational resonances of metal inks deposited on substrates
  - The vibrational resonance frequency is governed by the film/substrate adhesion strength
- A laser ultrasonics-based methodology will be developed as a non-destructive post-process control for assessing film/substrate adhesion quality in advanced manufactured sensors.



Preliminary ultrasonic waveform measured on an Ag film deposited on an alumina substrate



Accomplishments: Creep test rig

- Tested Creep Test Rig with SS 304 sample at 2500 psi and 300 ℃ in autoclave
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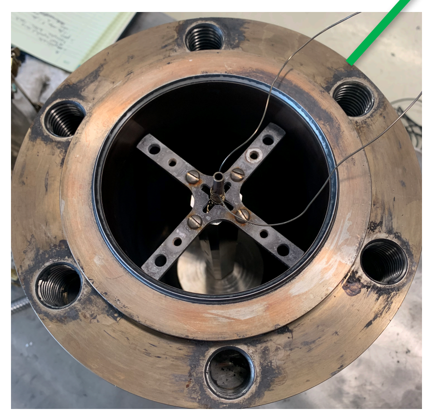
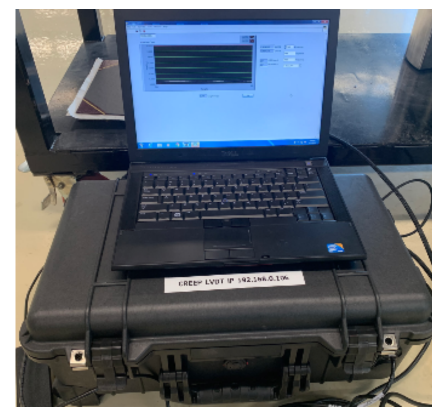
Static Autoclave System

SS 304 sample

Creep Test Rig post autoclave testing



Creep Test Rig inside autoclave



NI data acquisition system

Conclusion

- FY20 work included
  - Cross-cutting advanced manufacturing development (modeling/simulation, laser-based adhesion measurement)
  - Melt wire development and validation
  - Out-of-pile creep rig testing
- FY21 will focus on
  - Bi-metallic melt wire ink
  - Continued adhesion measurement development
  - Develop of real-time, in-pile test rigs for structural materials testing
- Questions?
- For addition questions or follow up discussion, contact me at: michael.mcmurtrey@inl.gov