INL/MIS-22-68287-Revision-0

Combinatorial and High-Throughput Materials Synthesis to Advanced Nuclear Materials Discovery

August 2022

Jason L Schulthess, Klint Stephens Anderson, Cody Hale, Matthew M Arrowood, Thomas L Maddock, Joseph Newkirk

Changing the World's Energy Future

INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance, LLC

INL/MIS-22-68287-Revision-0

Combinatorial and High-Throughput Materials Synthesis to Advanced Nuclear Materials Discovery

Jason L Schulthess, Klint Stephens Anderson, Cody Hale, Matthew M Arrowood, Thomas L Maddock, Joseph Newkirk

August 2022

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

July 13, 2022

Jason Schulthess[1]
Senior Staff Scientist

INL/MIS-22-68054

Automated Mechanical Testing System

Collaborators: Nathan Petersen[1], Phil Petersen[1], Mike Heighes[1], Sriram Praneeth Isanaka[2], Frank Liou[2]

1- Idaho National Laboratory
2- Product Innovation and Engineering, LLC

INL is managed by Battelle Energy Alliance for the US Department of Energy

Idaho National Laboratory

Outline

- Motivation
- Challenges and Opportunities
- AMTS

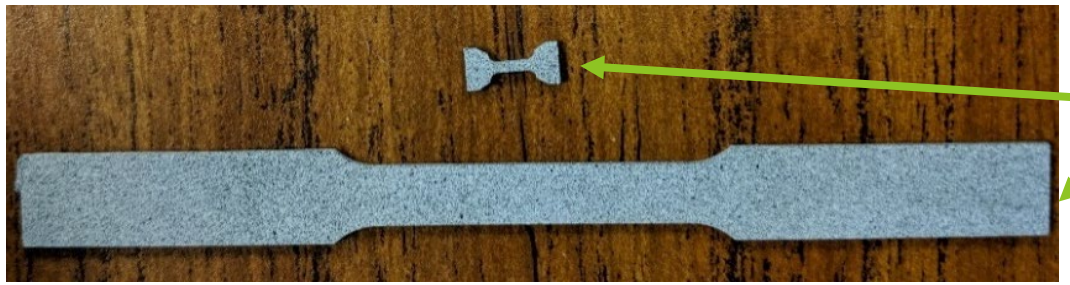
Motivation

- Need for structural materials for advanced reactor systems[1, 2, 3]:
  - Higher Energy Neutron Spectrum
  - Higher Temperatures
  - Corrosive Environments
- Research on new materials is being conducted, including on Multi-Principle-Element-Alloys (MPEA's, aka High-Entropy-Alloys, aka Complex-Concentrated-Alloys)
  - In a 2021 review paper[4], ~4000 papers published on MPEA
  - Of those, only ~100 were focused on nuclear applications and were topical on fabrication, modeling, ion irradiation, and corrosion.
  - Very limited neutron irradiation data on MPEAs
  - Nearly limitless composition space for MPEAs

1 – https://doi.org/10.1016/j.matre.2021.01.002
2 – https://doi.org/10.1016/j.cossms.2016.10.004
3 – https://doi.org/10.1038/ncomms13564
4 – https://doi.org/10.3390/e23010098

Challenges and Opportunities

- How to increase throughput, reduce cost, and increase data extracted from each specimen?
  - As of 8 July 2022, 1,683 entries in the NSUF Material Library containing the word “Tensile” in the sample description
  - Future experiments expected to generate large quantities of specimens of reduced sizes[1]
  - Automate mechanical testing,
  - Incorporate advanced Digital Image Correlation Analysis
    - Localized Strain Mapping
    - Spatial variation
    - Inhomogeneous materials
    - Etc.



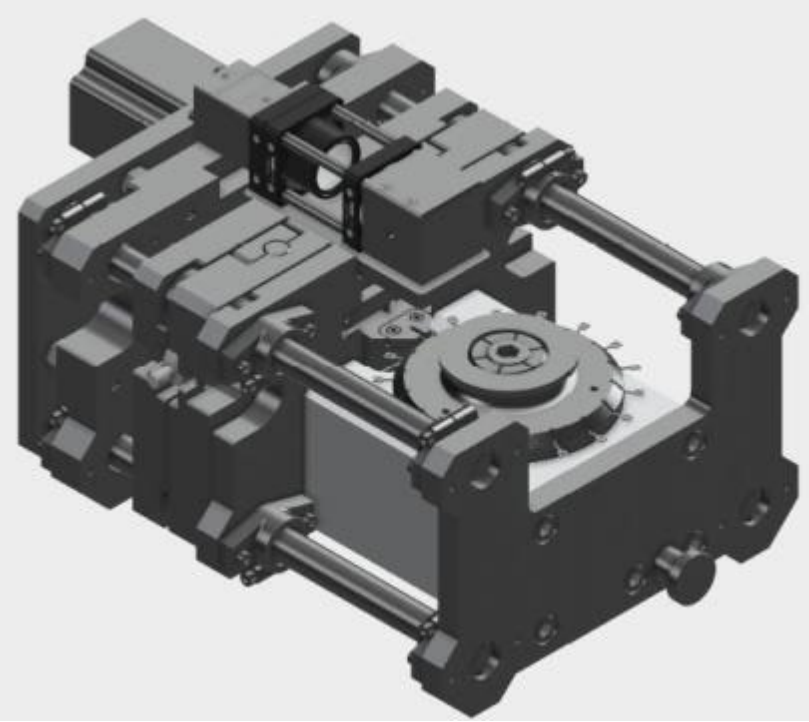
Miniature “MT2” Tensile Specimen
ASTM E8 Standard ”Sub-sized Specimen”

1 – Karnati, Isanaka, Zhang, Liou, Schulthess, “A Comparative Study on Representativeness and Stochastic Efficacy of Miniature Tensile Specimen Testing”, Accepted.

AMTS – Automated Mechanical Testing System

- Design driven by the following objectives:
  - Cartridge to hold multiple specimens
  - Automate test process
  - Accommodate small scale specimens
  - Incorporate advanced DIC
  - Automate data analysis
  - Small footprint to limit required space in hotcell

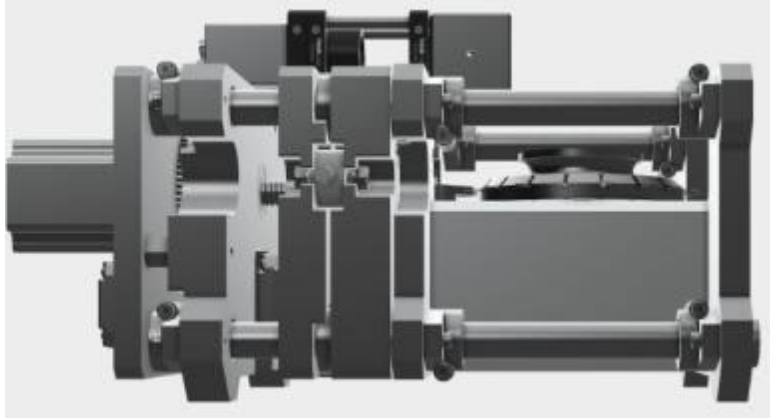
AMTS - Current design



Cartridge assembly has only 3 components

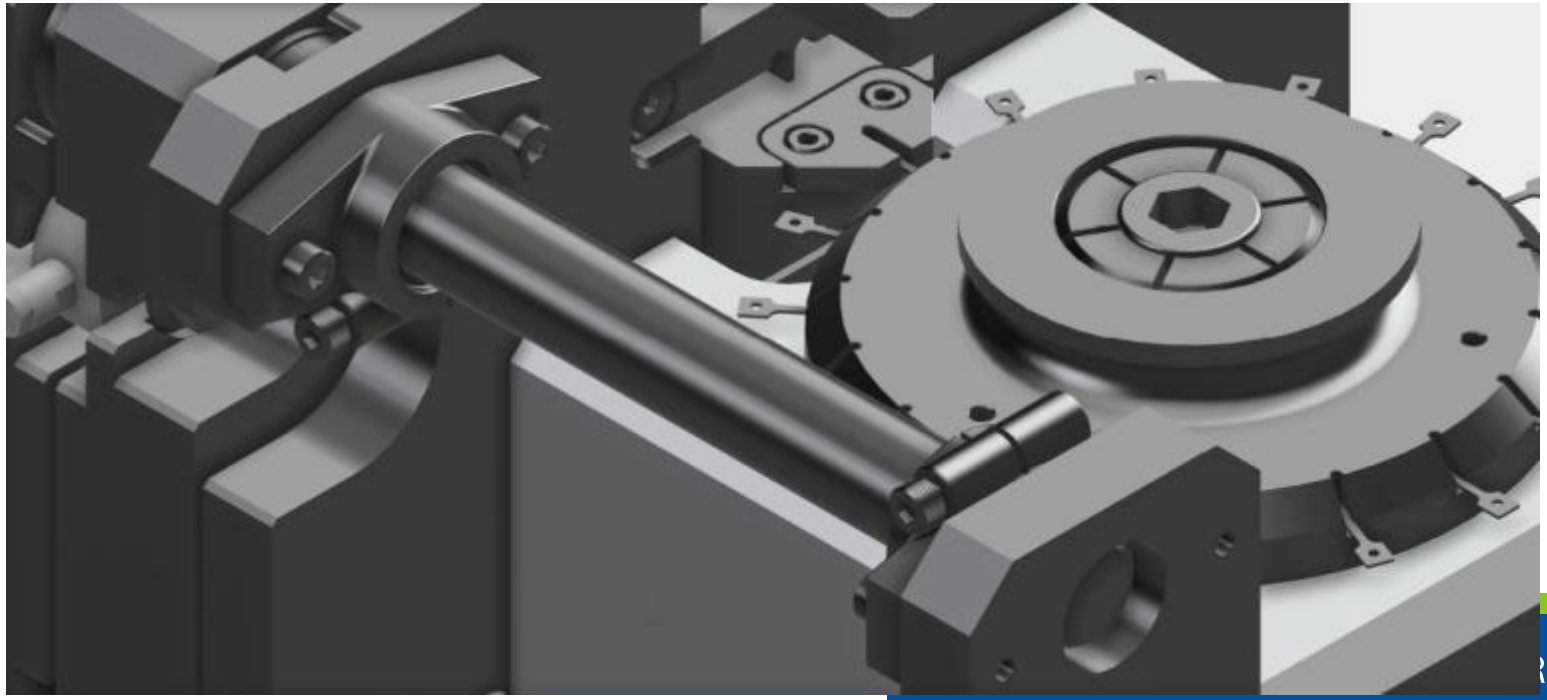
Size and weight accommodate hotcell installation

Cavity space and designed to collect and retain broken specimen



Electronics like motors, loads cells, camera assembly do not have direct line of sight with irradiated specimen

Number of specimen on cartridge is 24



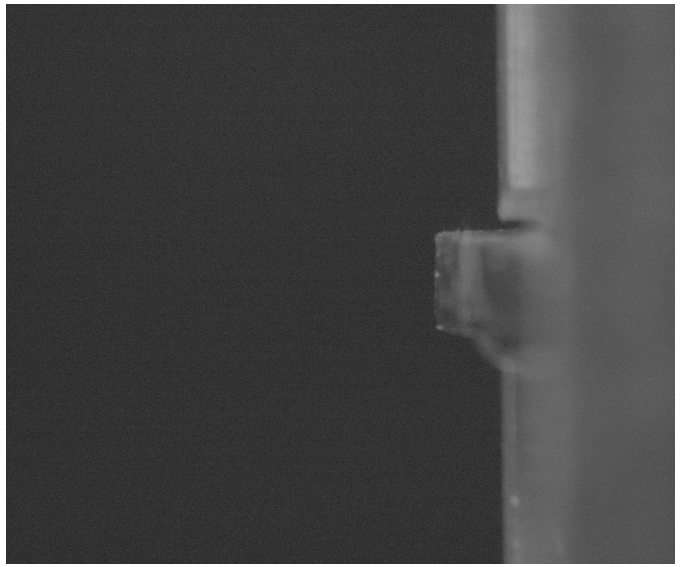
AMTS - Current design

This system weighs just over 30 lbs.

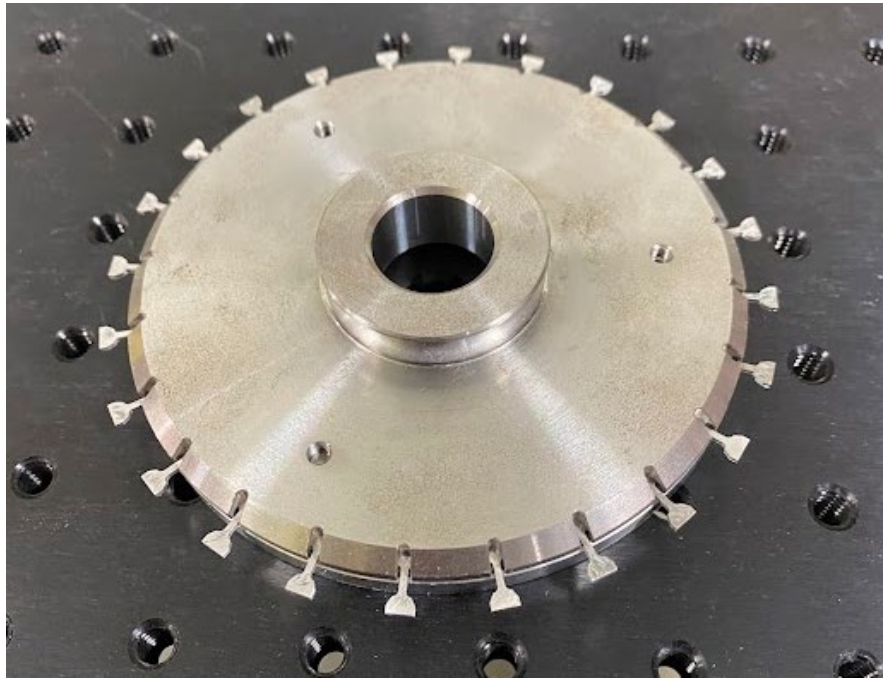
Footprint size is less than 2'*1'.

Cartridge has only 3 major components. Loading/unloading specimen designed for hotcell remote manipulators.

Swap cartridges for rapid testing or different specimen geometry.

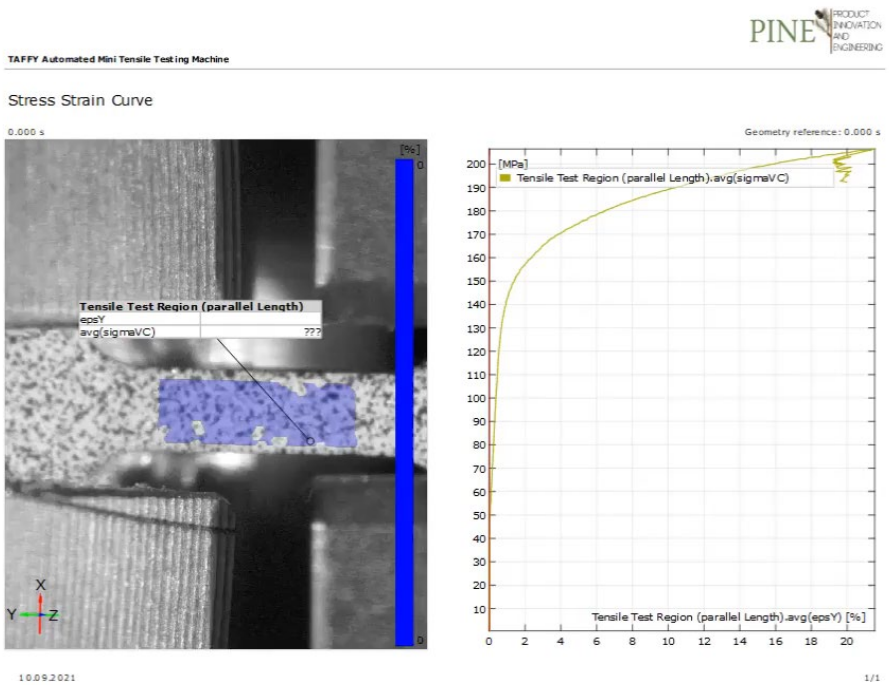


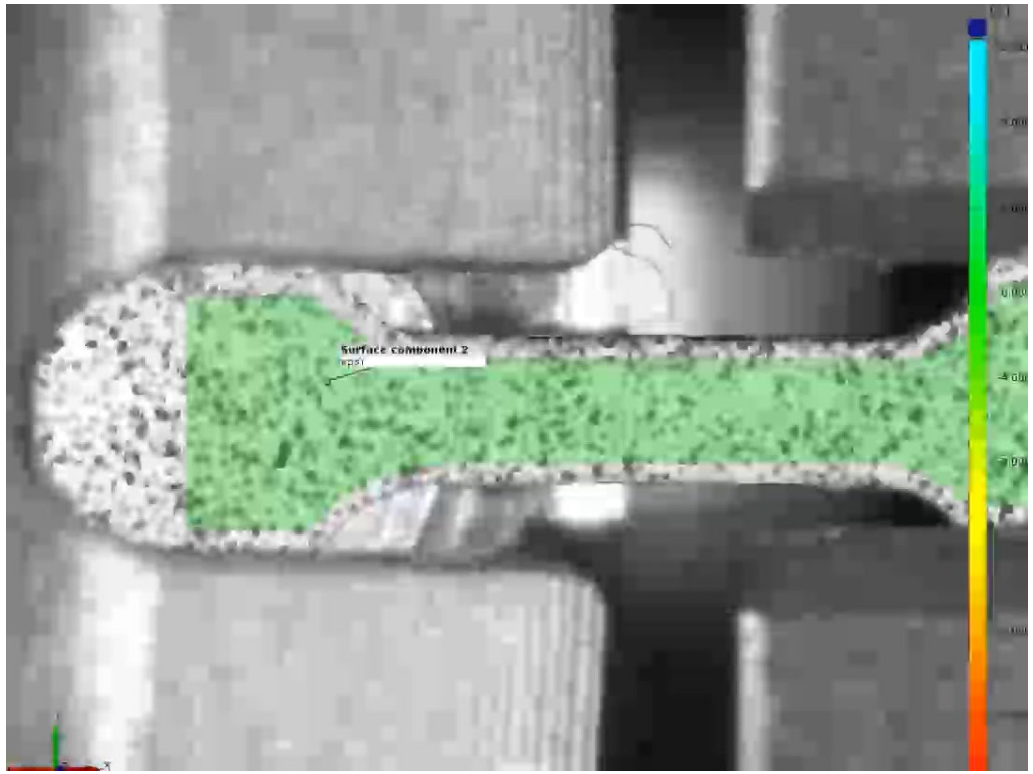
AMTS - System status



System at INL, working through functional testing and preparing for installation in hotcell.

AMTS - Software and data analyses

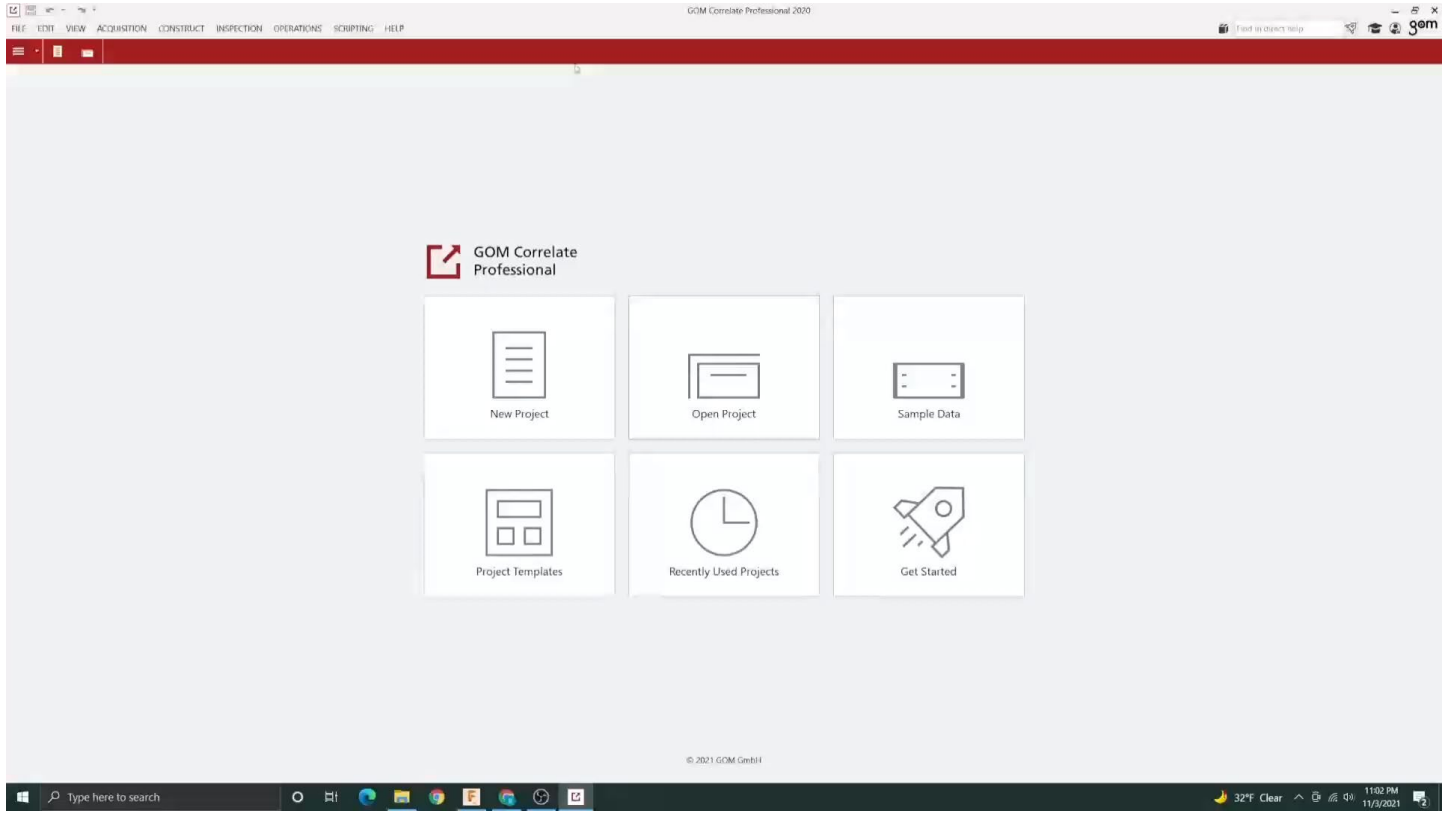




To facilitate automation and ease in data analysis, we chose an off-the-shelf software that offers scripting features; GOM Correlate

Written our own script to automate data assimilation from load cell and DIC to output the important material and test metrics as shown

AMTS - Automation through software



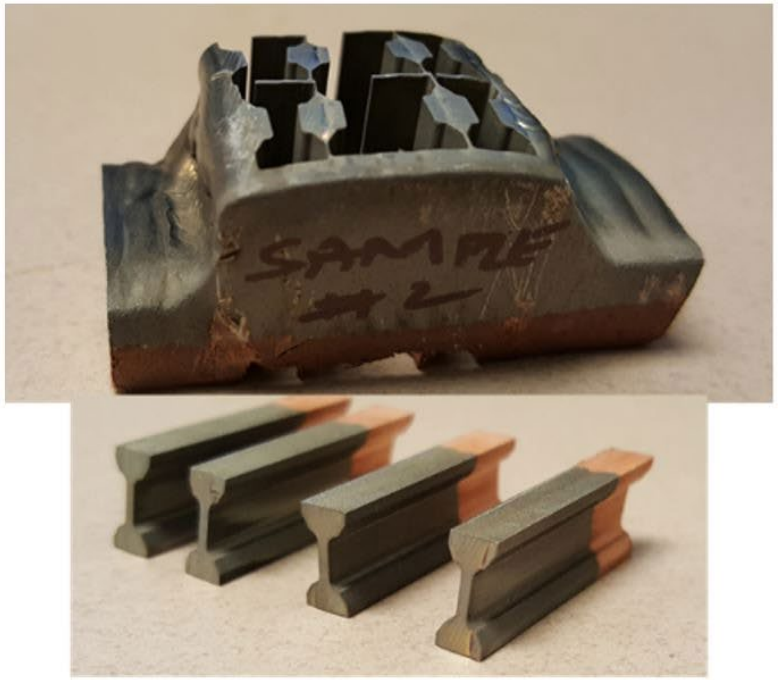
Utilizing scripting features offered by GOM Correlate to improve data sampling and analyses automation

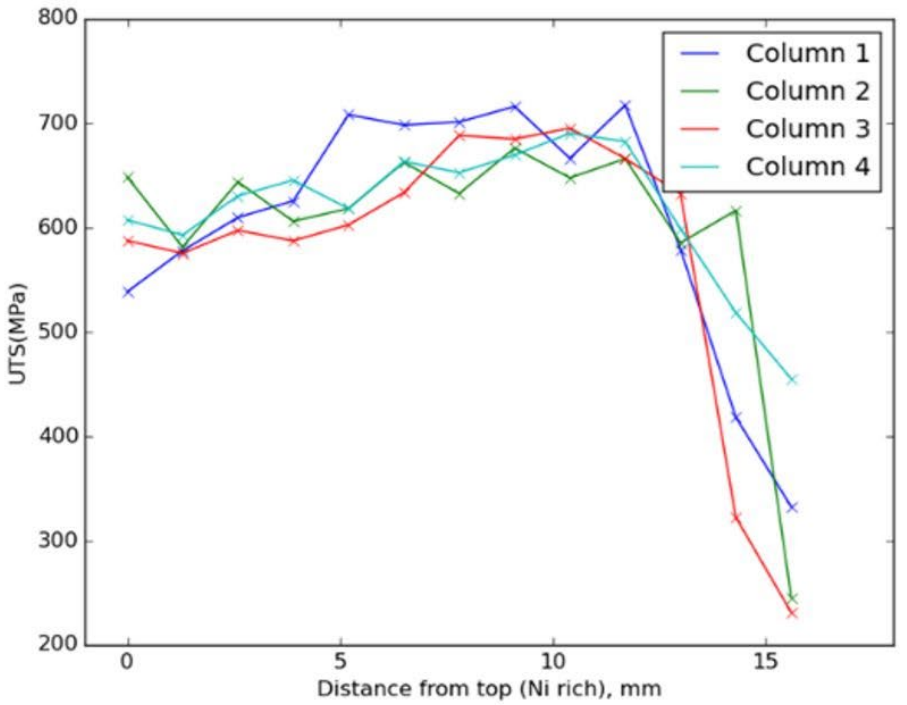
Current operation shown in video

Script provides an intuitive and interactive way to analyze and output the important data

Working on minimizing the amount of user interaction needed such that data sampling and analyses is also streamlined and automated

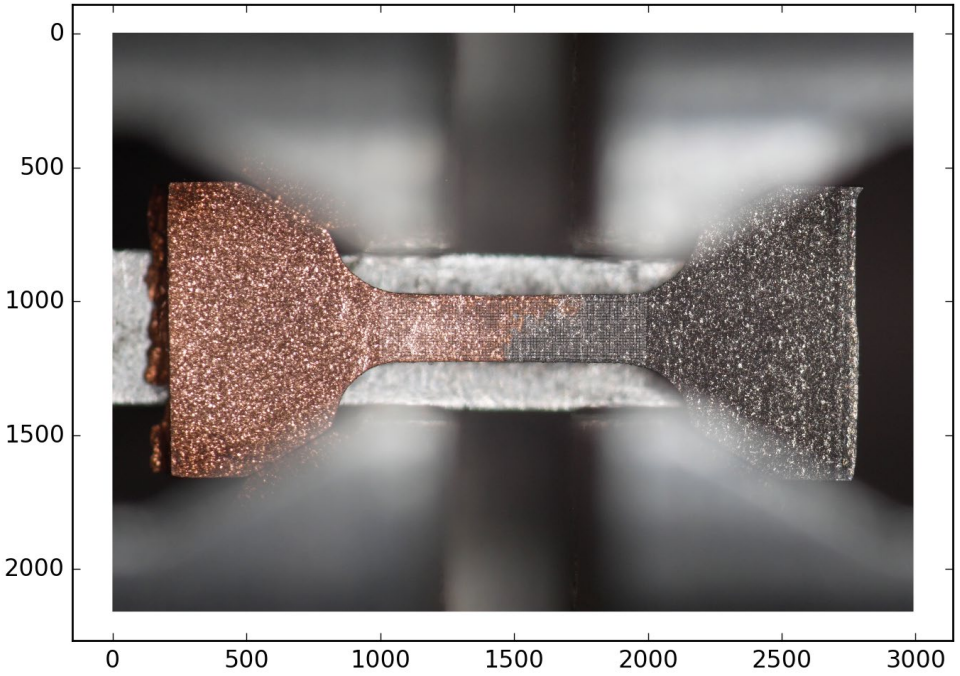
Potenial use: Spatial property characterization

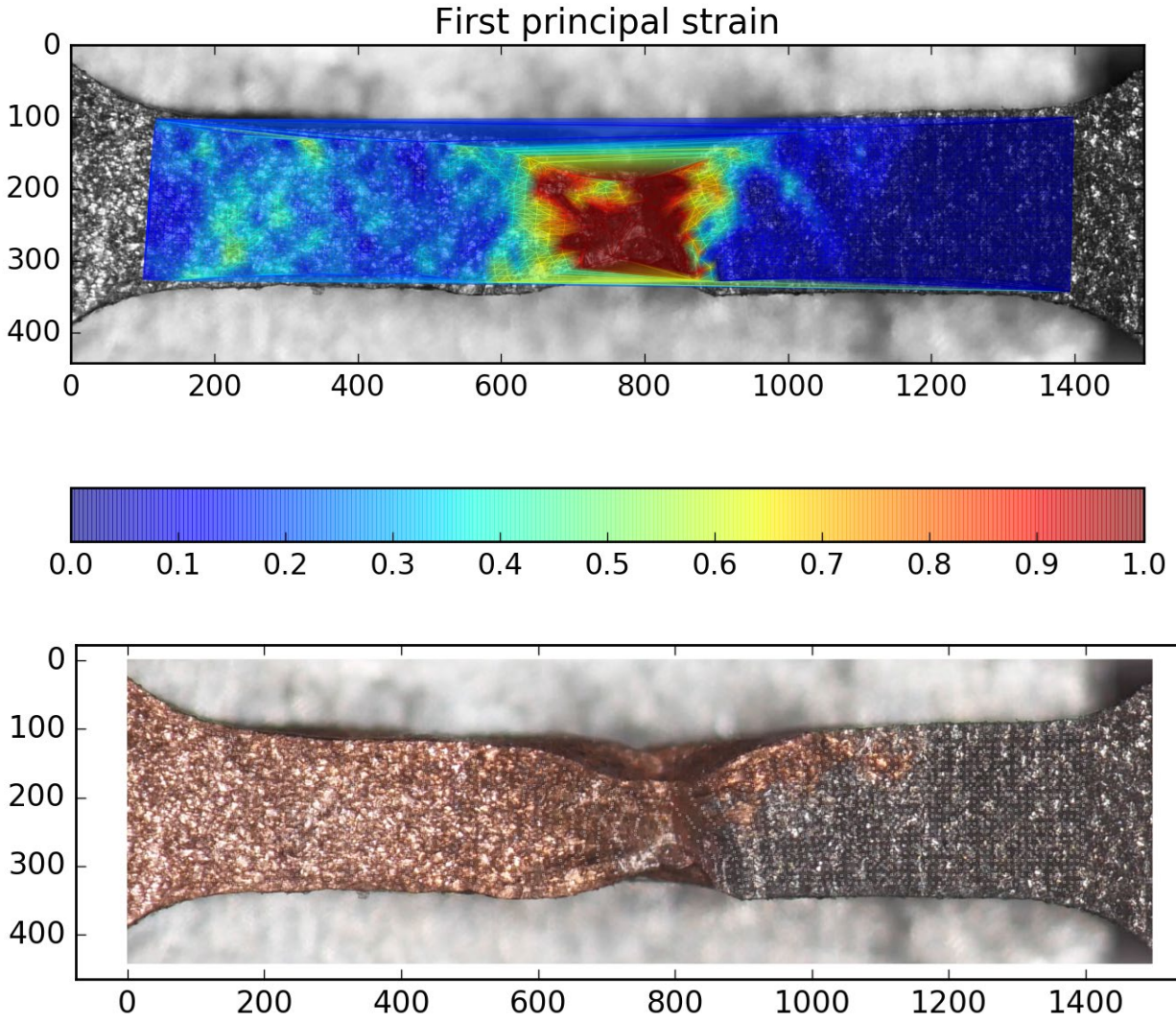




- FGM – Functionally Graded Material
- Each specimen different chemistry
- Variation along gradient, such as damage gradient across reactor pressure vessel wall

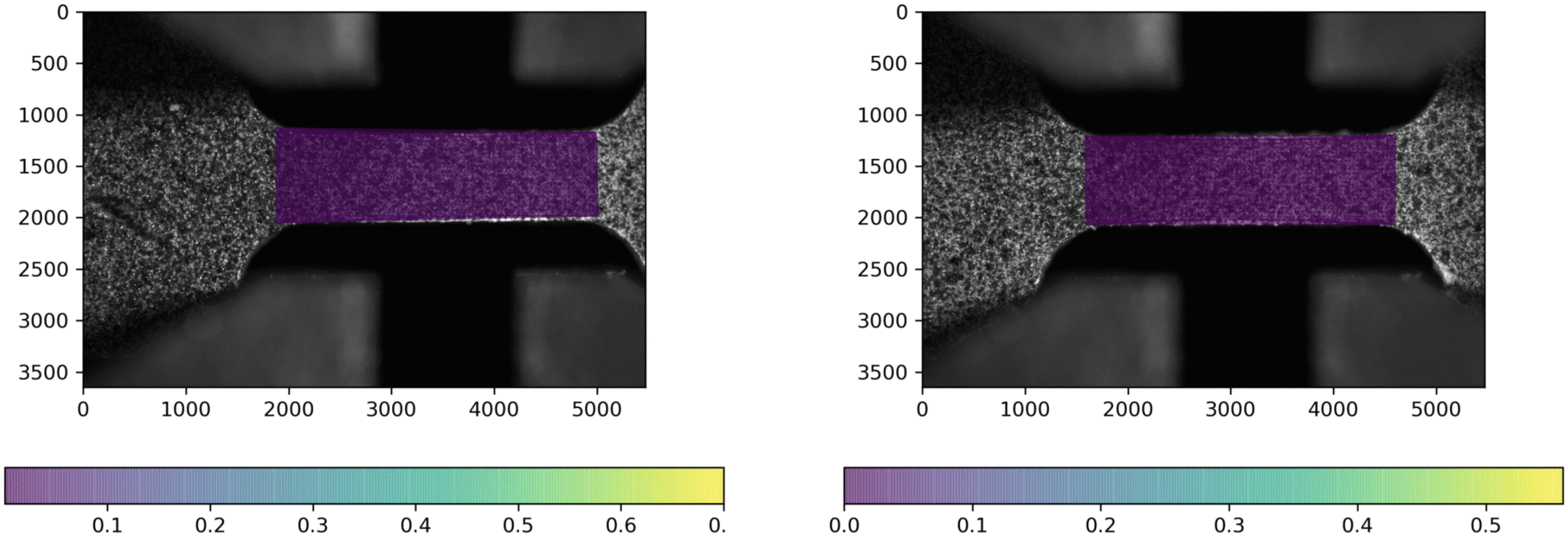
Inhomogeneous material testing





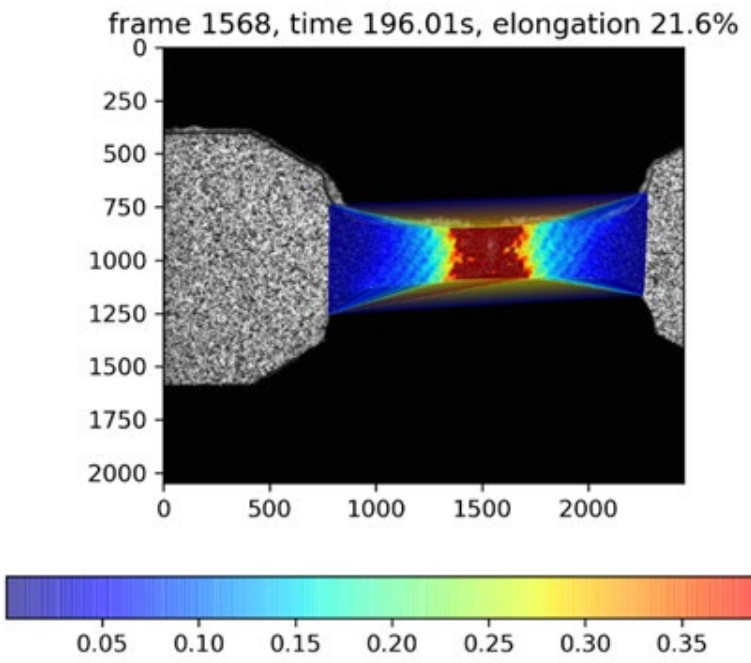
- Cu to Inconel 625 FGM
- Inhomegeneity in materials will lead to failure in the softer material (copper) during tensile testing which was visually captured using the AMT's Digital Image Correlation (DIC) system

Potenial use: Strain Evolution

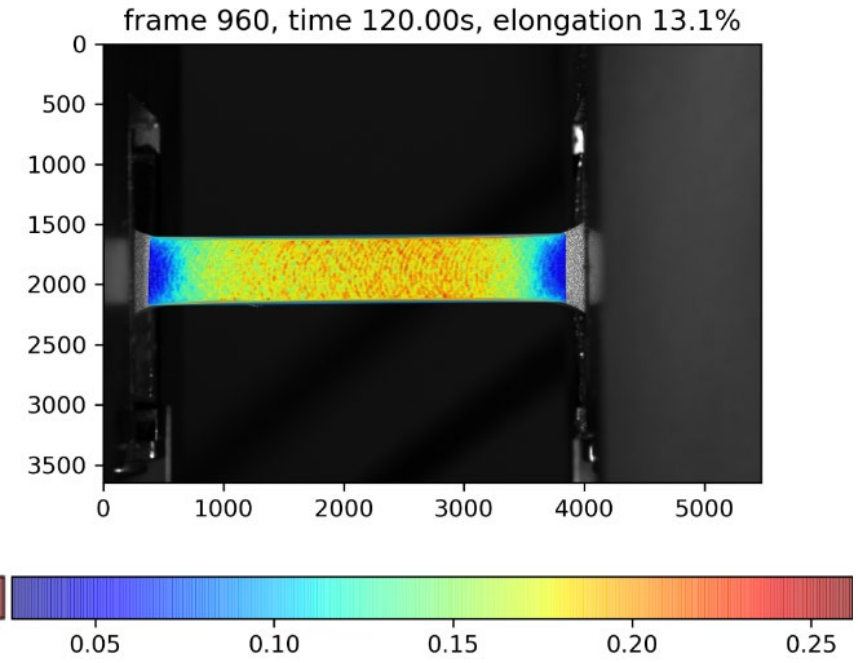


- Inhomogeneous strain distribution

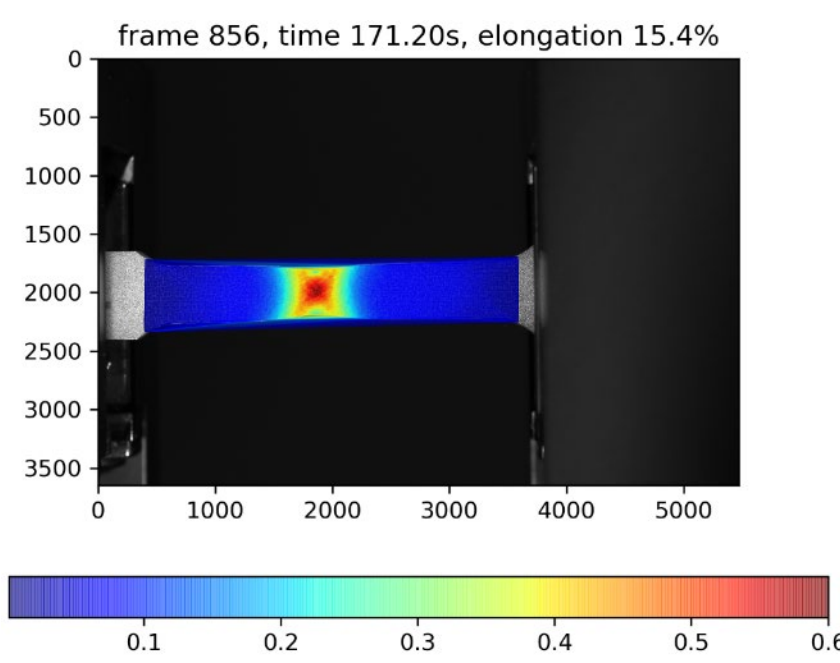
Different Materials and Phenomena



SS304L



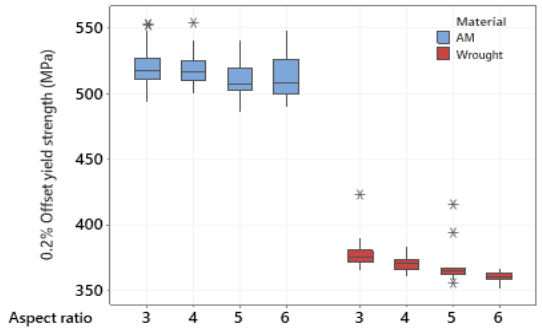
Ti – Strain field

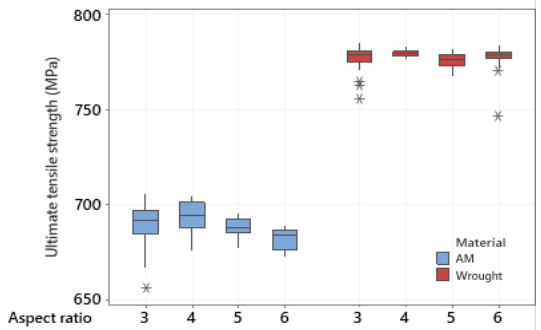


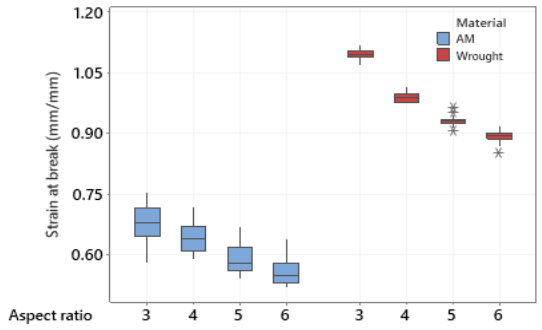
Al 6061-Before Failure

- Effects of strain hardening from CNC machining in Ti strain field image,
- Strain field development in different materials
- Effects observable irrespective of specimen size or material type

Impact and benefits

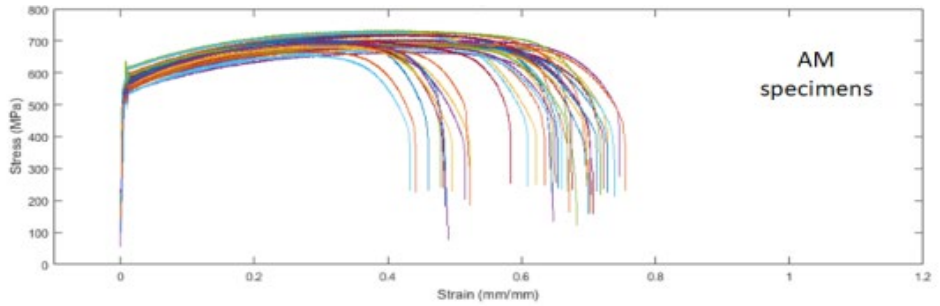


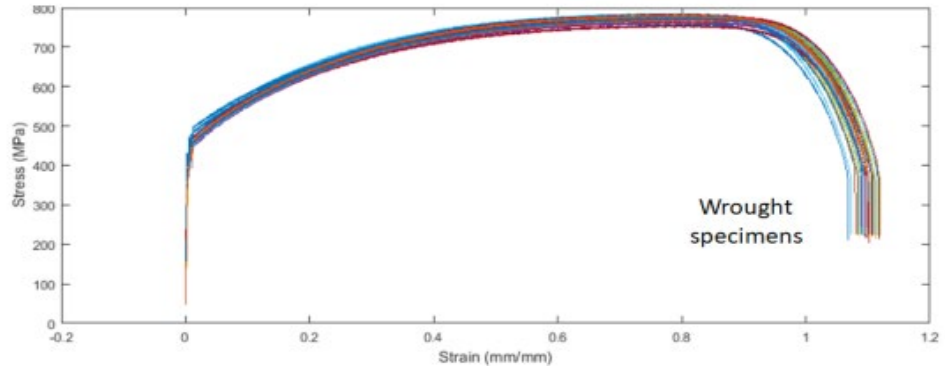




Number of valid measurements

	Strain at break		Ultimate tensile strength		0.2% Offset yield strength	
Aspect ratio	AM	Wrought	AM	Wrought	AM	Wrought
3	27	30	32	30	38	30
4	30	22	25	21	34	22
5	30	25	24	25	36	25
6	23	27	18	27	26	27





The ability to rapidly acquire 100's-1000's of data points could have significant scientific and societal ramifications

Scientific: Modern materials and manufacturing processes exhibit critical flaw sizes, inhomogeneity, anisotropy, and other effects that are best identified by miniature specimen and high throughput testing

Societal: Gathering large amounts of data to provide statistical relevance and confidence especially when working with expensive or hazardous materials

Idaho National Laboratory

Battelle Energy Alliance manages INL for the U.S. Department of Energy's Office of Nuclear Energy. INL is the nation's center for nuclear energy research and development, and also performs research in each of DOE's strategic goal areas: energy, national security, science and the environment.