Advanced Materials Characterization and Nuclear Product Detection for LENR

Robert V. Duncan, Ph.D.
President's Distinguished Chair in Physics and Professor of Physics
Texas Tech University

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Robert V. Duncan, Principal Investigator, Texas Tech University

Technology Summary

Category B project to support all ARPA-E LENR Experiments

Exceptional custom nuclear products detection (^4He, ^3He, ^3H, n, γ) beyond the commercially available state of the art

Materials and nanoparticle fabrication, visualization, and analysis with experimental integration and ultra-fast data capture / analysis

Technology Impact

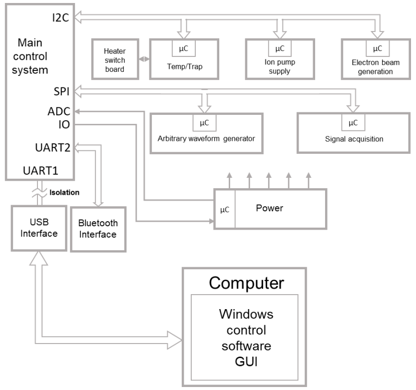
Wide, area AI detection of emergent LENR features at 20 nm resolution

Surface and sub-surface change detection with species / lattice identification

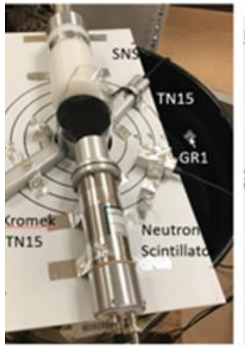
Calibrated nuclear product extraction with accurate measurements

Proposed Targets

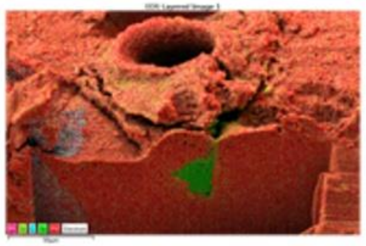
Metric	State of the Art
^3He, ^4He, ^3H production measurements for custom experiments	*Qualitative detection without calibrated extractions, at ~nM level (We resolve sub-pM with calibrated extractions)*
Materials visualization with AI change detection	*Non-existent today at nanoscale resolution (We feature this today)*
Ultra-fast data capture	*Usually ~ 50 ksps (We are > 50 Msps)*

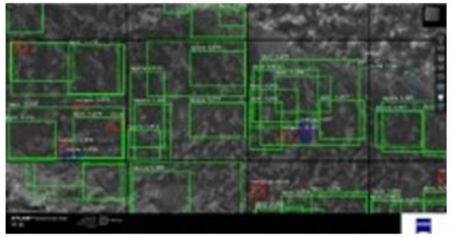


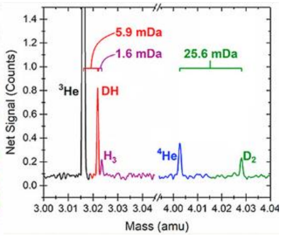
New design philosophy using 'smart' modules to implement each major function. Each module is autonomous, and process control and measurements are made at the module.



Calibrated particle detection and spectroscopy with experimental customization, and fast noise rejection







Beyond state-of-the-art commercial measurements with custom experimental integration

All with proven ultra-fast data capture, secure storage, and analysis tools

CHANGING WHAT'S POSSIBLE

We don't speculate, we innovate and measure!

Hypothesis

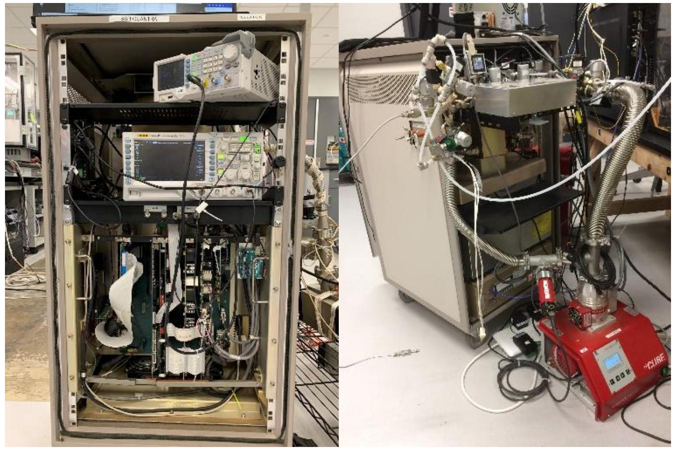
INVESTIGATIONS WITHIN THIS EXLPORATORY TOPIC REQUIRE VASTLY IMPROVED, CUSTOM-DESIGNED MEASUREMENT & EXPERIMENTAL FAB SYSTEMS TO DETERMINE THE PHYSICS UNDERLYING LENR PHENOMENA

- Quantitative 'nuclear ash' measurements at < pM levels / excellent linearity
- Materials fabrication with before / after characterization, change detection
- Focused Ion-Beam (FIB) surface nanofabrication
- FIB for sub-surface structure and dynamic event reconstruction imaging
- Custom electronic designs for event-triggered data capture and storage
- Ultra-fast electromagnetic spectroscopy for event characterization
- Multi-physics and nuclear transport modeling and simulations
- Nuclear radiation measurements and advanced design capabilities
- Machining, 3D printing, and advanced metal joining capabilities

Experimental Capabilities

- Custom Ion Cyclotron Resonance (ICR) mass spectrometry to detect ‘nuclear ash’
- Advanced fabrication, nanofabrication and nanometer-scale imaging
  - Zeiss Crossbeam 540 SEM, SE, EBS, EDS, EBSD, CVD, and FIB system with robotic scan and Atlas mosaic software capabilities
  - Custom AI system to detect multi-scale emergent phenomena and statistics
  - Access to complete measurement suites of TEM, XRD, XRF, SIMS ICMS, and other imaging diagnostics within Engineering and Geosciences
  - Extensive analytic chemistry and materials analysis capabilities
  - Full machine shop, orbital welding, and 3D printing capabilities
- Advanced electronics and fast data capture capabilities
  - Ultra-high frequency measurement and spectroscopy to 110 GHz
  - Event-triggered data capture and storage on RAID drives at 50 Msps
- Nuclear radiation sensing, detection, and spectroscopy with design capabilities
- Vastly improved open-system calorimetry free from prior systematic errors

‘Nuclear Ash’ detection at unprecedented sensitivity and stability



Achieved mass resolution better than 0.0001 Da at mass-3 with excellent stability

Detection of He in ICR is difficult due to collisional de-population at its very high ionization potential (24.6 eV)

Upgraded manifolds allow separation from carrier gases

Originally, commercial instrument bounded by 5 MHz data collection in the A/D converter (Nyquist limit of 2.5 MHz).

→Corresponds to m/z ~ 5.6 Da, For us, 2 Da operations: excite f = 6.91 MHz

Higher frequencies provided by a Rigol arbitrary waveform generator.

Also upgraded various electronics for better customization of controls

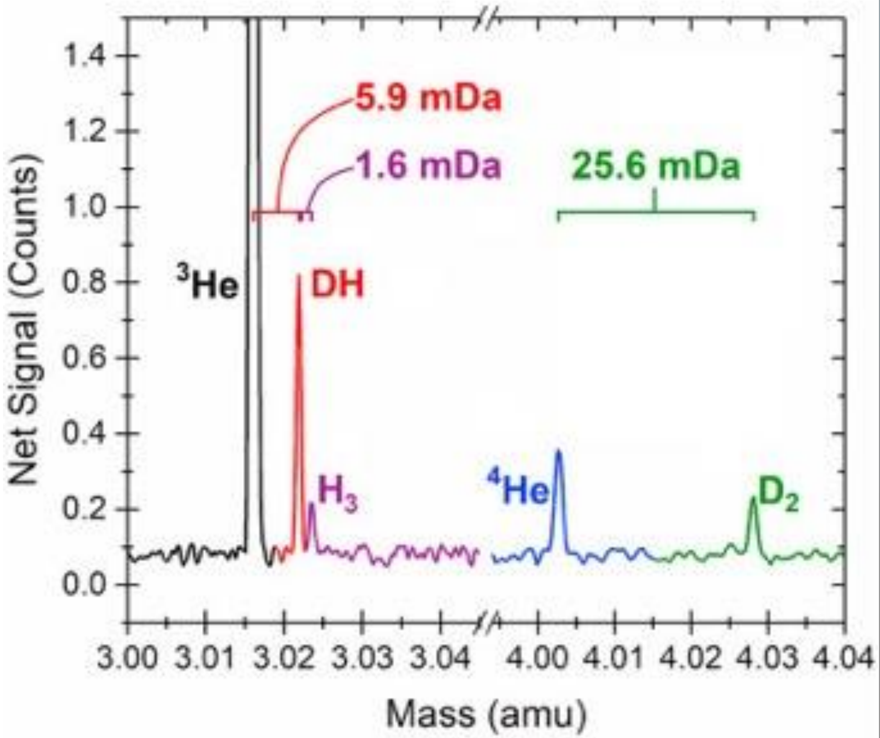
- We quantitatively measure and distinguish between ^3He, ^4He, tritium, D_2, DH, DT, and isotopes associated with sample air invasion
- Simultaneously detects the co-resonating species ^{20}Ne, ^{40}Ar as an indicator of real-time sample air invasion
- Measurement environment around instruments: [^4He] < 0.02 PPM
- Linear measurements between ~ 0.5 pM to 50 pM
- Calibrations (twice weekly) demonstrate excellent stability (~ 5% / week)
- Tritium may be detected as T^+, HT^+, DT^+, H_2T^+, etc., but scintillation detection of tritium’s low-energy beta is far better

Perkin Elmer’s Quantulus achieves ~ 10 fM sensitivity to T, with excellent linearity and specificity

We have this capability within CEES, and advanced tritium extraction techniques

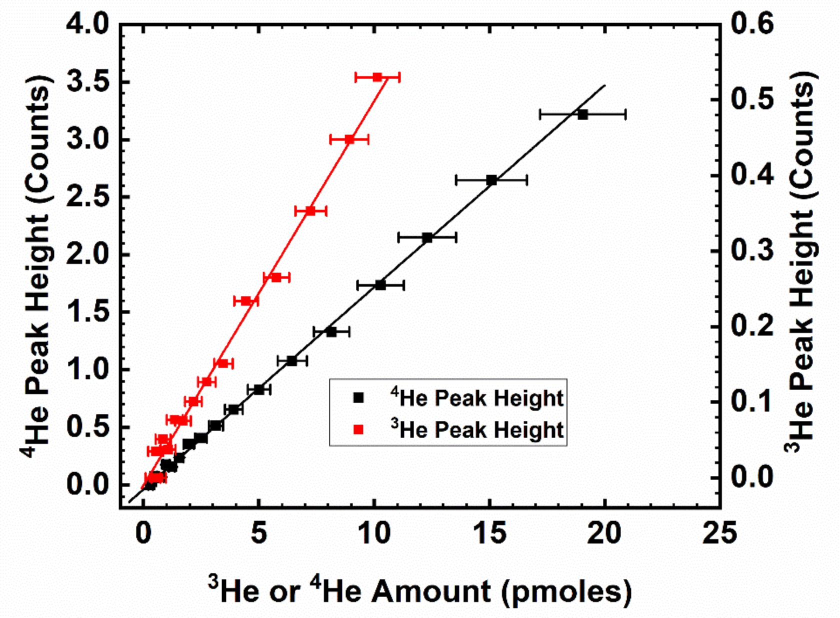
- Upgrade to I2C electronic control and measurement architecture

Integrated smart sensors result in an adaptable, ultra-fast smart instrument!



High-Resolution FT-ICR Mass spectrometer: LLOQ < pMole

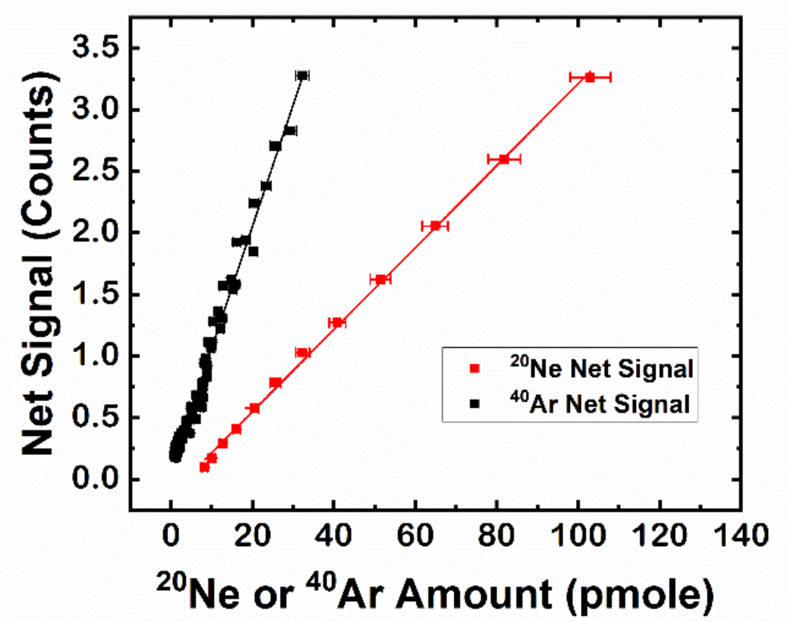
^{4}He & ^{3}He Calibration Curves



Excellent linearity from noise level (~0.24 pM) to 20 pM in ^{3}He and ^{4}He measurements.

Corresponds to a sensitivity near one joule of excess heat, if one ^{4}He atom is produced for each d+d fusion event that releases 23.8 MeV of energy

^{20}Ne and ^{40}Ar Calibration curves



- The ^{4}He and ^{3}He calibration gases had concentrations of 6.52 and 5.76 ppm in a D_2 matrix, respectively.
- LLOQ = 0.555 picomole ^{4}He.
- LLOQ = 1.16 pmole for ^{3}He.

- ^{40}Ar: 5.00 ppm, ^{20}Ne: 4.24 ppm, both in a H_2 matrix

This new ability to detect air invasion in real-time (^{20}Ne and ^{40}Ar measurements) & ability to sample gas from wet cells while eliminating magnetron motion has major benefits for the LENR community, especially for labs studying the vapor headspace of experimental cells.

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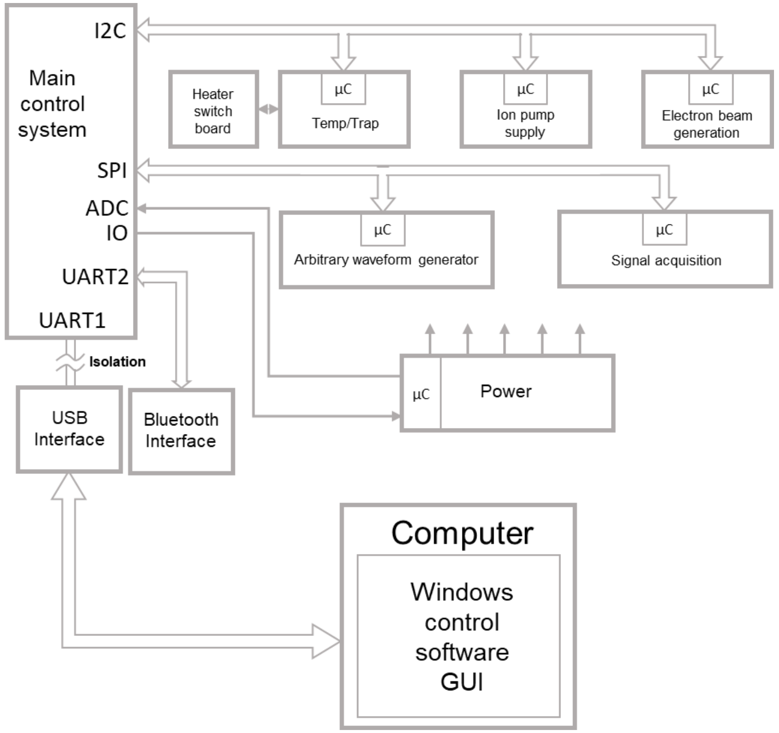
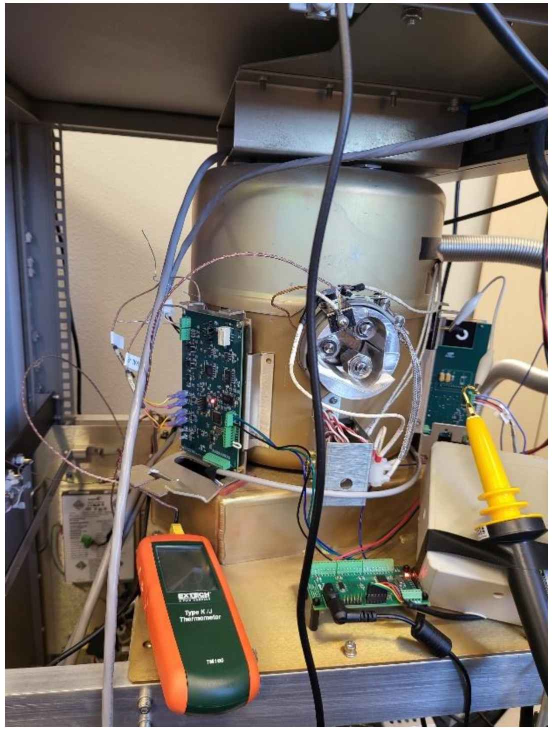
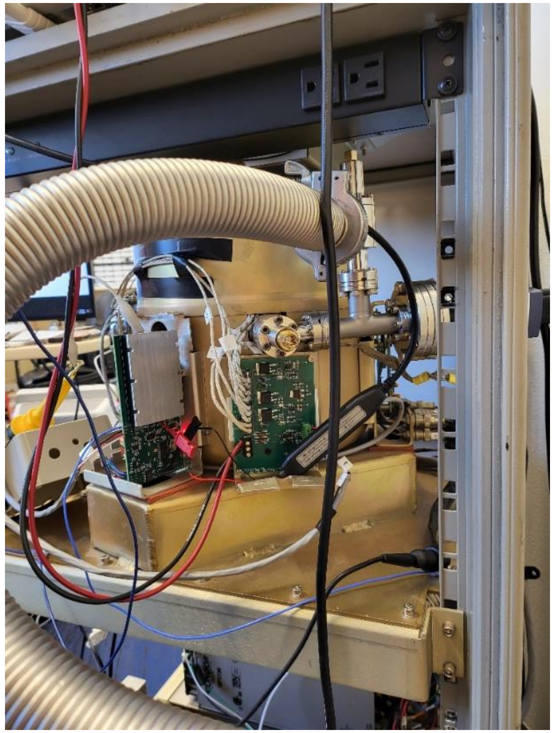
High-Resolution FT-ICR Mass spectrometer, Upgrade



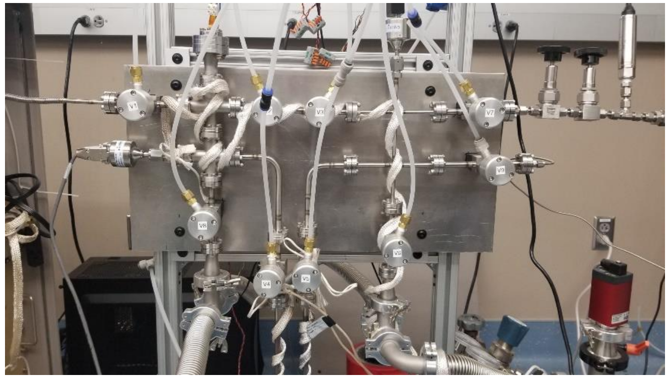




Rebuilding a 1990's-era FTICR-MS originally produced by Siemens, and expanding its range down to 2 Da. New design architecture uses 'smart' modules to implement all major measurement functions. Each module is autonomous, and process control, optimization, and measurements are made at the module.

Gas Extraction

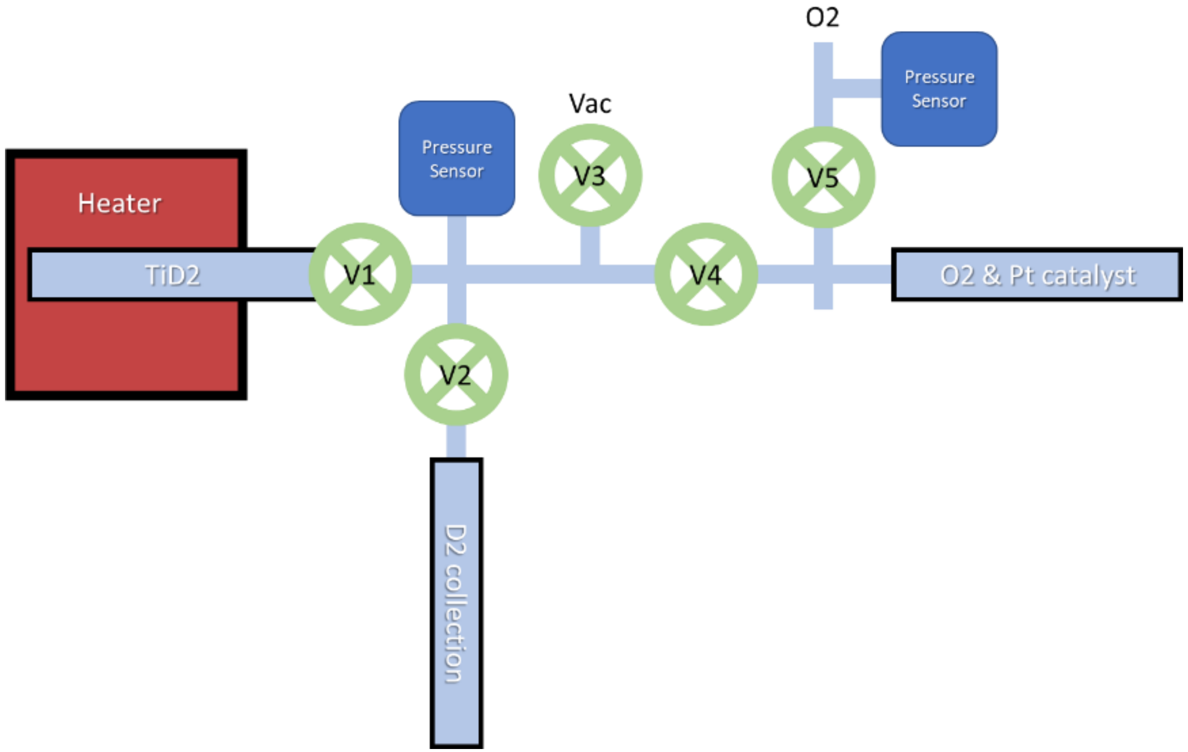
Orbital Welding Advances



We successfully developed a bellow valve system utilizing CF flanges through orbital welding. This system offers replacement flexibility while providing big advantages, such as ultrahigh vacuum compatibility, and the ability to undergo extreme bake-out processes. Compared to VCR fittings, it offers greater rigidity, reliability, and hermeticity that vastly surpasses Swagelok compressed fittings and Nupro bellows valve leak-through rates. To our knowledge, no other provider offers valves of this nature. Furthermore, these valves can be utilized in absolute helium-leak-free manifolds, and other ultra-critical performance applications as well.

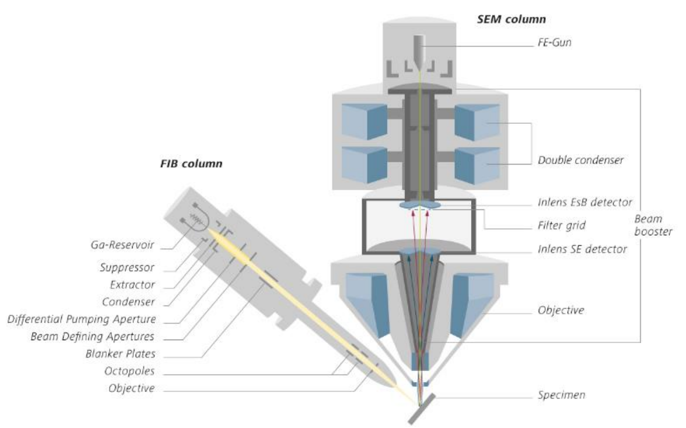
Gas Extraction (Con't)

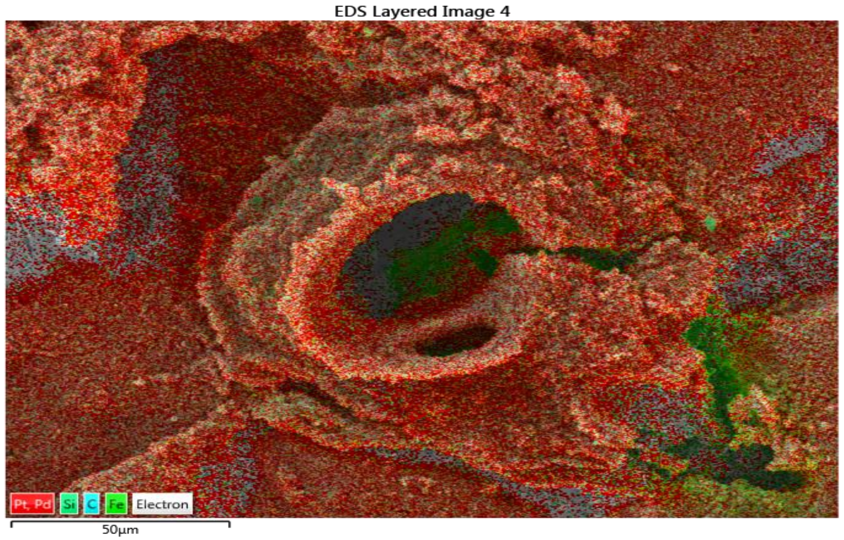
Advanced hydrogen and helium isotope extractions

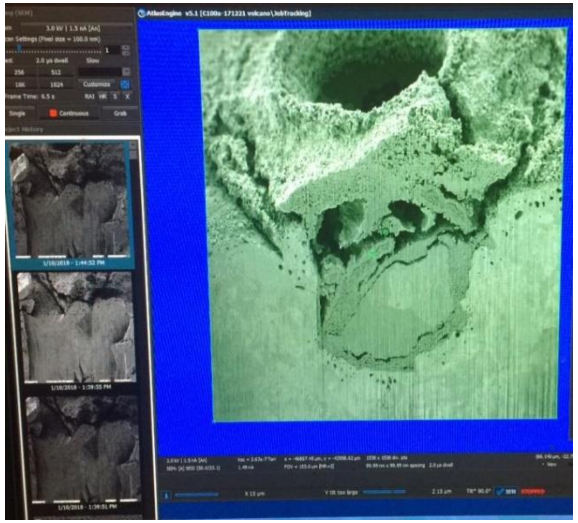


- We determine the tritium extraction efficiency using knowledge of the initial hydrogen isotope loading levels
  - Close collaboration on the experimental design is essential
- Differentiate between D_2, H_2, HT, DT, etc., when necessary, using FTIR
- We know how many metals and technical materials retain atmospheric He, how to extract He, and its recharge rate

Electron Microscope, Large-Area Imaging, and EDS mapping



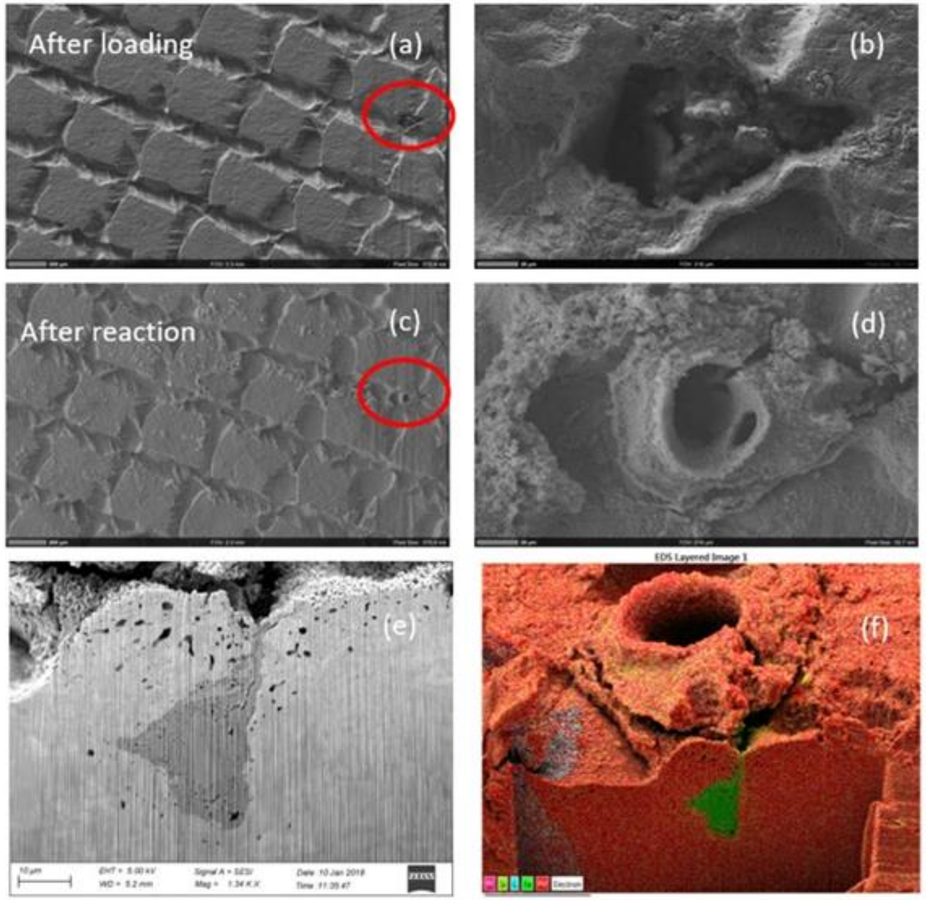




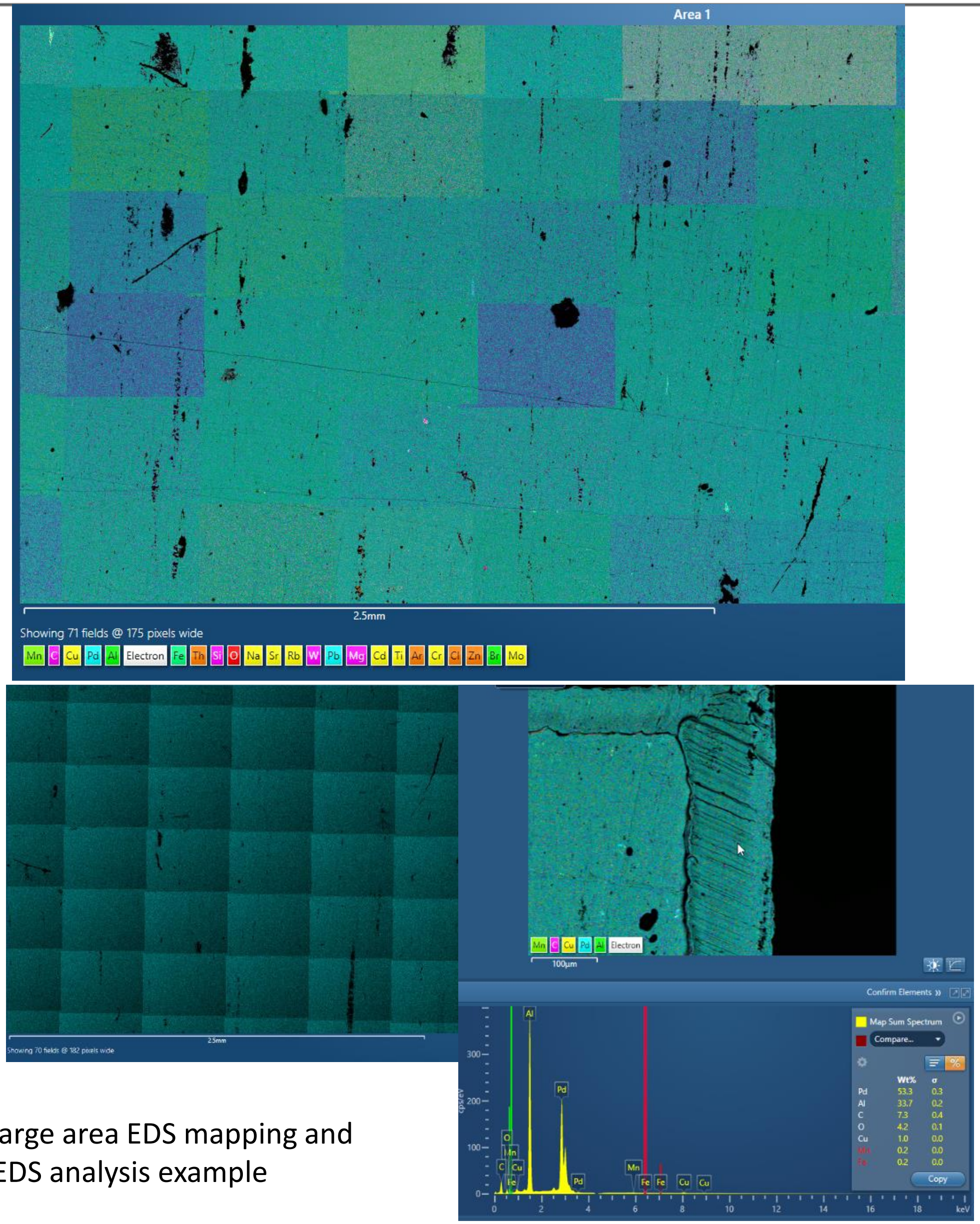
CEES capabilities include advanced metallurgy, orbital welding, 3D printing, machining, SEM / TEM imaging, FIB nano-milling, CVD, wide-area mapping of electrodes, electrode surface change detection, AI-based emergent feature detection in materials / statistical analysis, and physical and chemical characterization technologies (XRD, XRF, EDS, EBSD, etc.)

Includes a Zeiss 540 Cross-Beam Imaging / FIB System:
< 20 nm imaging / elemental analysis / structure res, 50 nm FIB cut-away resolution across entire cathode
Hitachi TEM capabilities, and collaborations with MURR for reactor and cyclotron irradiations and NAA

Electron Microscope, Large-Area Imaging, and EDS mapping (Con't)



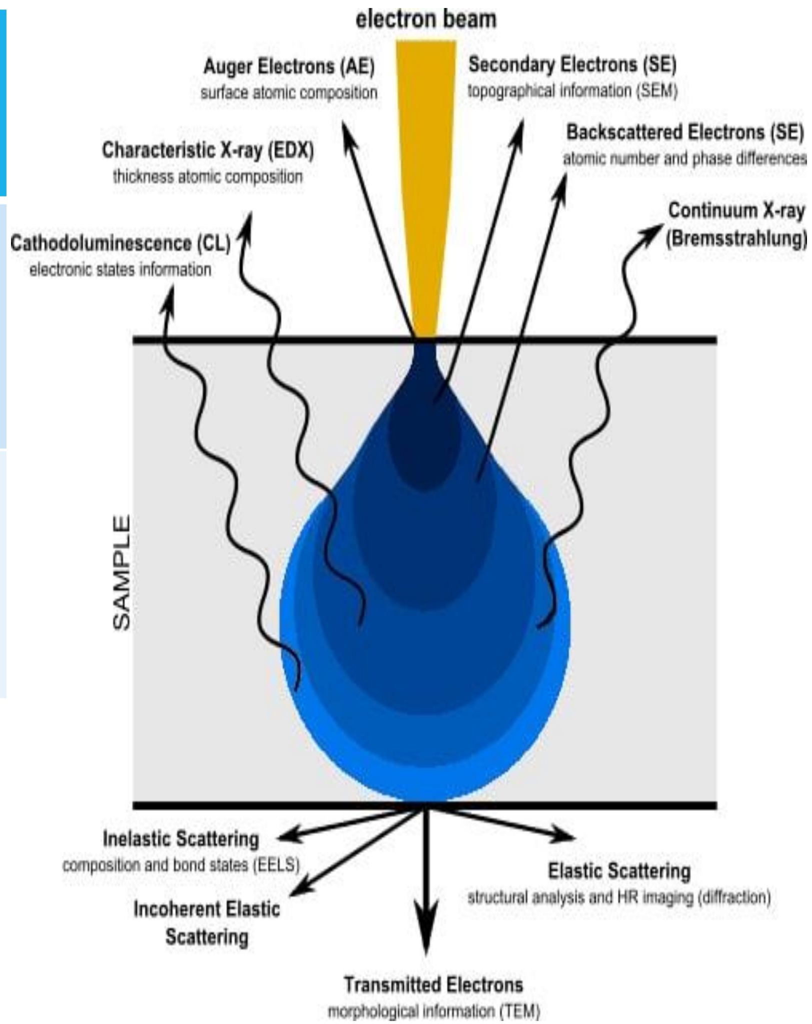
Large area mapping of entire cathode after loading; (a,b): zoom in comparison of the volcano feature after reaction (c,d,e,f); BSD image of volcano after FIB cutting (e). The EDS image in green indicates sub-surface iron (f) EDS study of the volcano following a FIB facing cut verifies the difference of chemical compositions, and the composition of the ejecta.



Large area EDS mapping and
EDS analysis example

Electron Microscope, Large-Area Imaging, and EDS mapping (Con't)

	Data collection High res	Data collection Low res	Operator Time	Data Acquisition
Large area SE/BSD imaging: 0.5 cm^2	@40 nm per pixel resolution: 8 h @20nm per pixel Resolution: 32h	@100 nm per pixel resolution: 3h	1h	automation
Large area EDS analysis: 0.5 cm^2	@500 nm per pixel resolution: 22h	@2 μm per pixel resolution: 3 h	0.5h	automation



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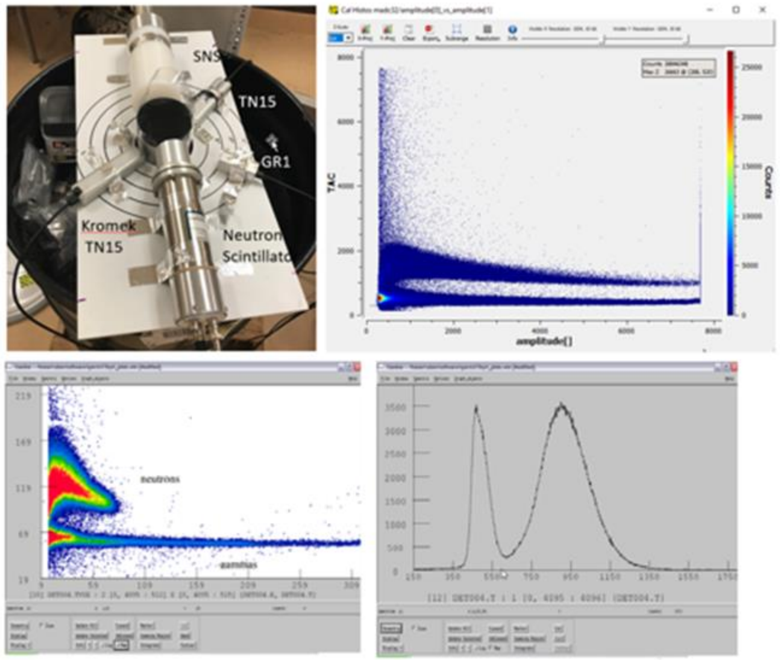
Nuclear Detectors and Calibration Sources

Nuclear Detectors, neutron, gamma



A typical radiation project at TTU (b) Time to Analog Converter (TAC) versus Energy in in 252Cf measurements made with the neutron spectrometer (c) TAC versus Energy plot for neutrons from the d+d → n+^{3}He reaction with a neutron energy of about 2.45 MeV. (d) TAC spectrum for neutrons from the d+n → n+3He reaction and background gamma radiation. The gamma peak is at the left; the neutron peak at the right.

Tritium Extraction & Detection

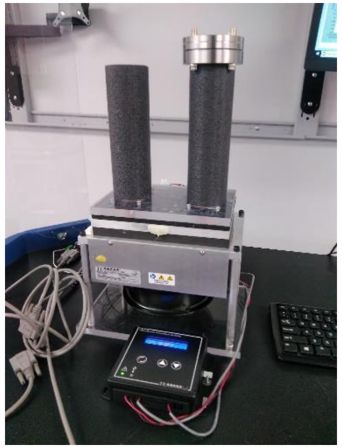
- Excellent T extraction and radiation metrology have been developed at TTU
  - Quantitative measurements of D_2 extraction efficiency from TiD_2 irradiated samples
  - Perkin Elmer Quantulus 6220 Scintillator system measured T produced with 10^{-14} mole accuracy, excellent calibration and linearity.

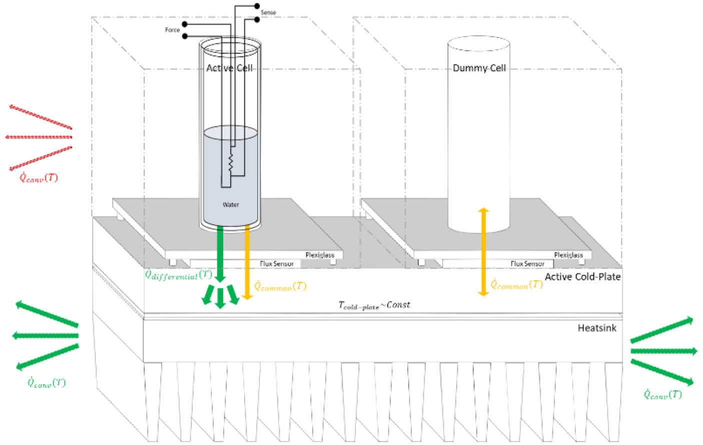
Calibration Sources: Cf-252, Cs-137, Co-60, AmBe, Thorium-241, Am-241(alpha, on the way)

Reactor and Cyclotron Collaboration with MURR (Columbia, MO).

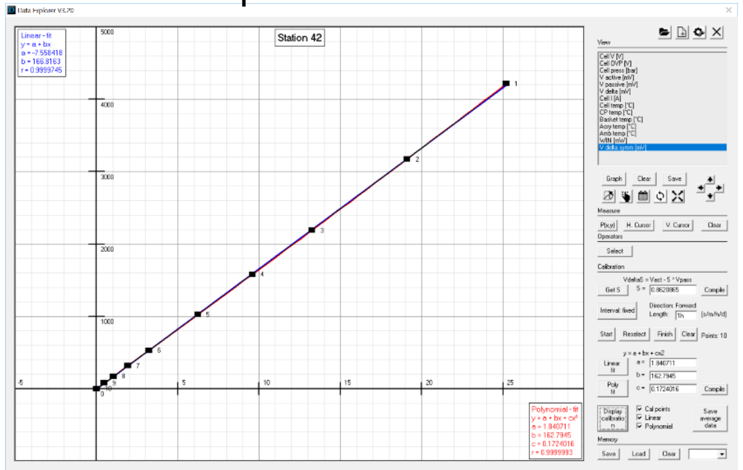
Open-System Calorimeters

Open-system, open-air, differential calorimeter



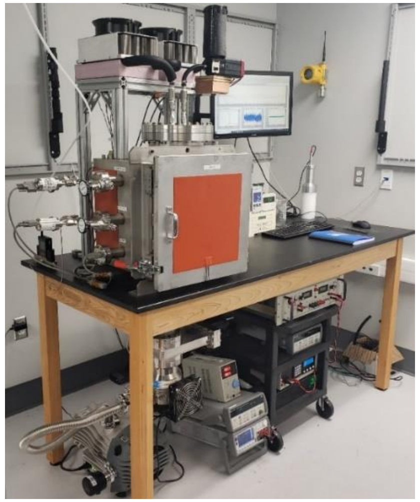


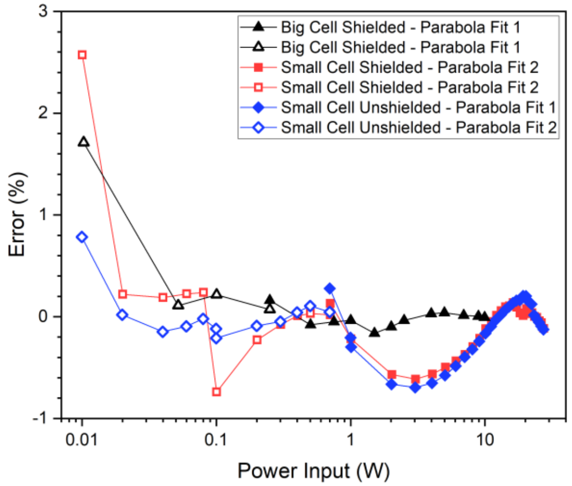
Based off of differential calorimeter using a heatsink, Redesigned using an thermoelectric isothermal cold-plate.



Custom GUI creates calibration curves with unique correction factors that compensate for sensor sensitivity and varying flow/loss paths.

Open-system, vacuum calorimeter





- Differential dual-stage thermoelectric module-based calorimeter
- Wide operating range of 1 mW up to 28 W, or 250 °C
  - Operation beyond 28 W/250 °C feasible, though untested
- Typical error < 1 % beyond ≈ 20 mW
- Heat transfer plate utilized to enable wide variety of cell configurations

References:
S Lacouture et al., Review of Scientific Instruments 91, 9, (2020) 095102
M Kelley, An Open-System Differential Vacuum Calorimeter, under Review

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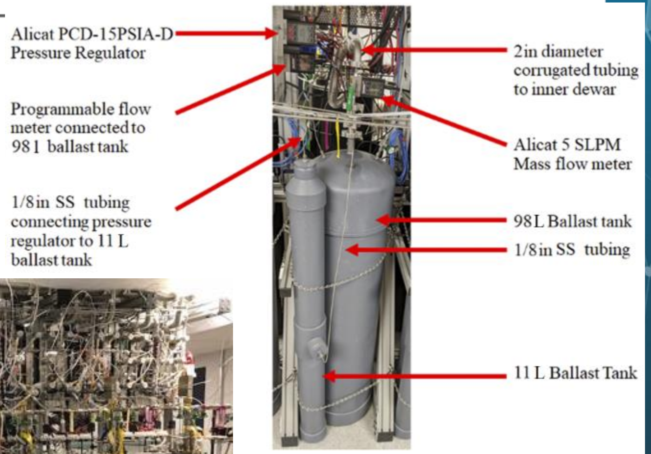
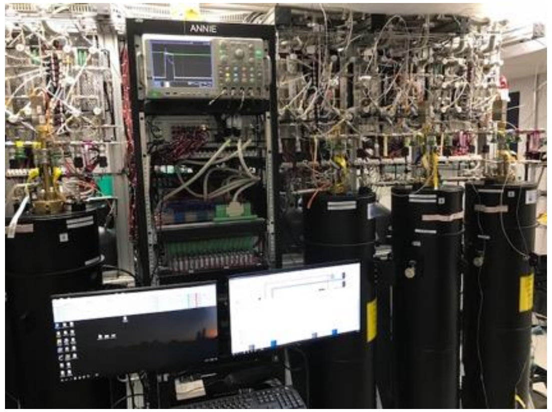
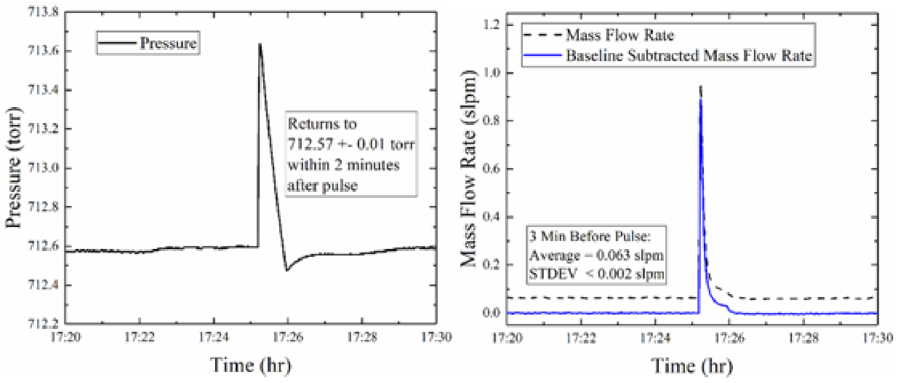
Open-System Calorimeters

Liquid nitrogen evaporative calorimeter

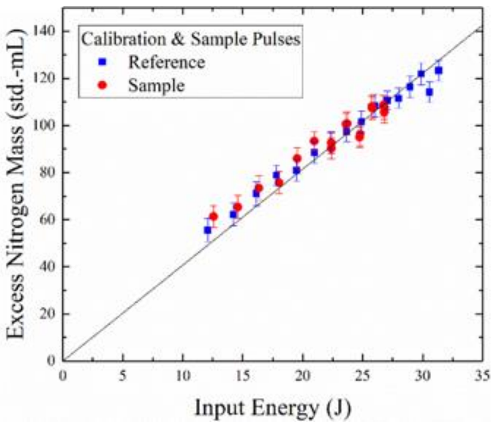
- Small changes in atmospheric pressure create large changes in the evaporation rate
  - LN_2 pressure must be maintained to one part in 10,000 continuously
  - Range: 1 to 50 J heat pulses, from 10 µs to 1 ms duration
  - As-built calorimeter is 96% efficient and very repeatable, with a calorimetric sensitivity of ~ 25 mW
  - Dewar was carefully designed to eliminate super-heating
  - N_2 mass flow resolution of 0.05 std. ml / s

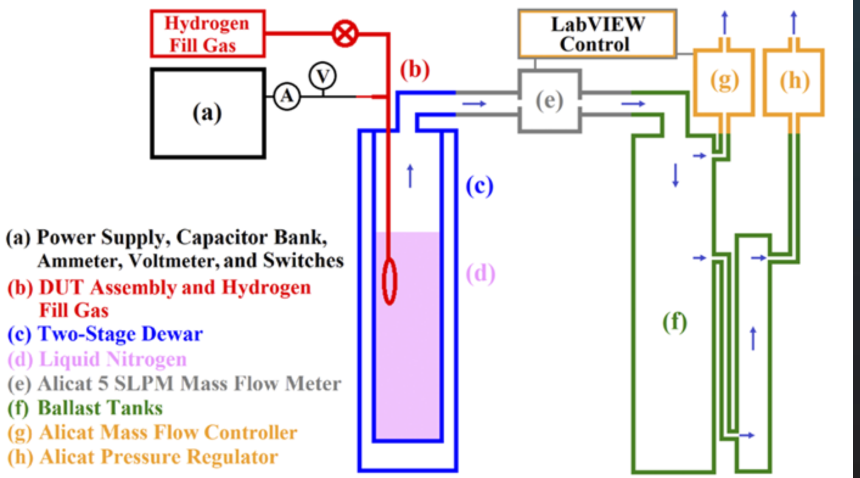






(a) (b)





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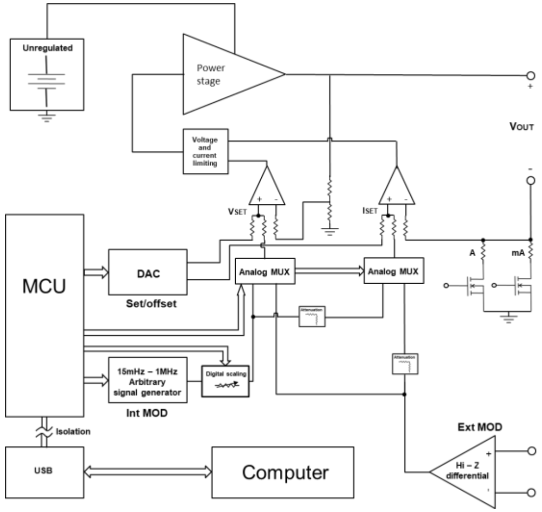
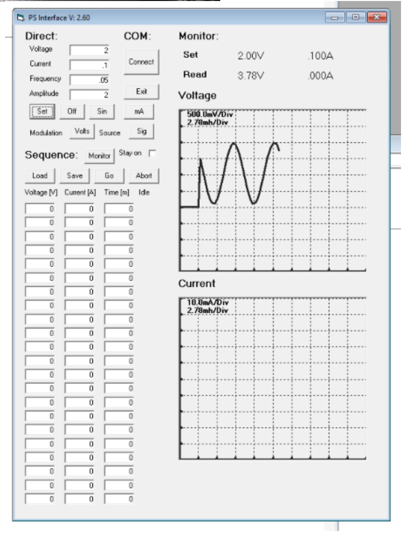
Andrew K. Gillespie, *et al*., Review of Scientific Instruments, 91 (2020), 085103

Custom-Engineered Electronic Systems

Low-noise, internally and externally modulated voltage and current channels / power supply.

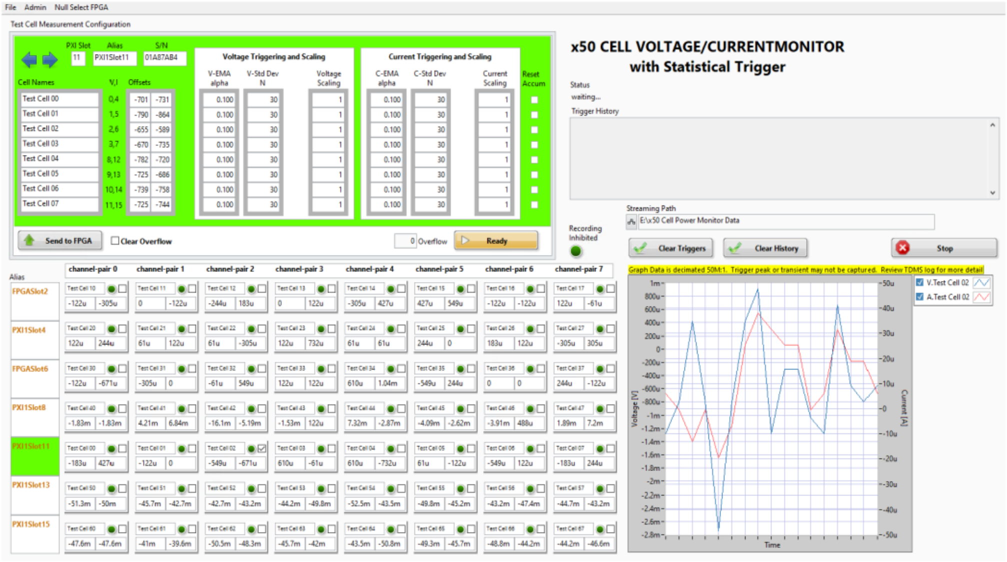




Low-noise, power supply, where voltage or current mode can be modulated by an internal signal generator, or external waveforms. Digitally controlled via a Windows GUI.

Statistically Triggered Voltage·Current Monitor

- High speed data acquisition for up to 56 channel pairs
  - Each channel pair sampled up to 50 kHz
  - Statistically triggered channels sample rate up to 50 MHz
- NI based system utilizing FPGA, digitizer, and 5.7 TB RAID drive
- Higher speed measurements obtained using 2 GHz oscilloscope
- EM radiation spectrum with remote mixers up to 110 GHz (W)



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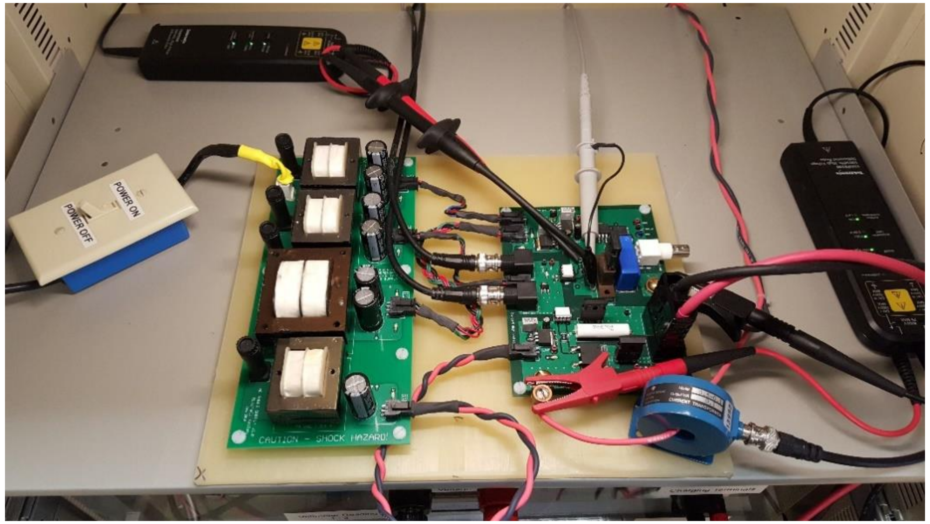
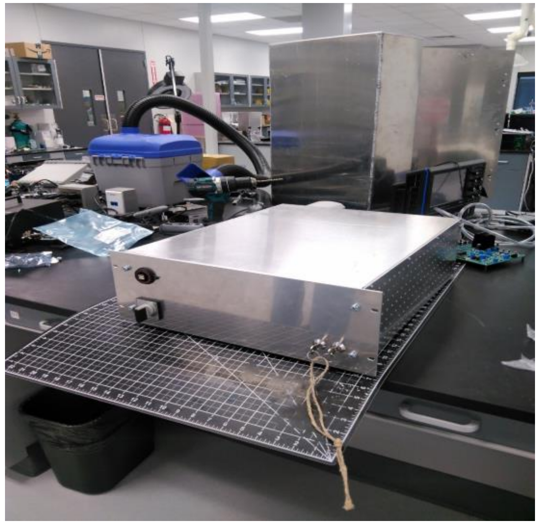
Engineered Electronic Systems --- Capability Gallery

Wide Bandgap Avalanche Sensor Test Bed (solid-state PMT)



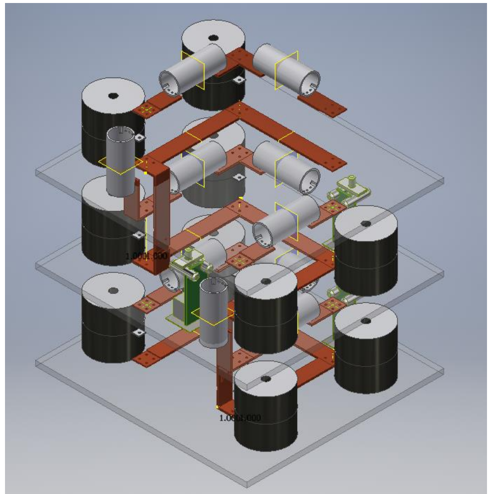
High-Current Digitally Controlled Pulser for High Current and Magnetic Field Applications



Low-Inductance, High-Power Pulse Test Bed

V to 500V,
I to 300A
Pulse duration
400 μs with only 2% drop
With full V, I pulse digitization

High-Power Transmission Line Pulser



4,000 V,
4,000 A maximum with 10 μs to 1 ms pulse duration

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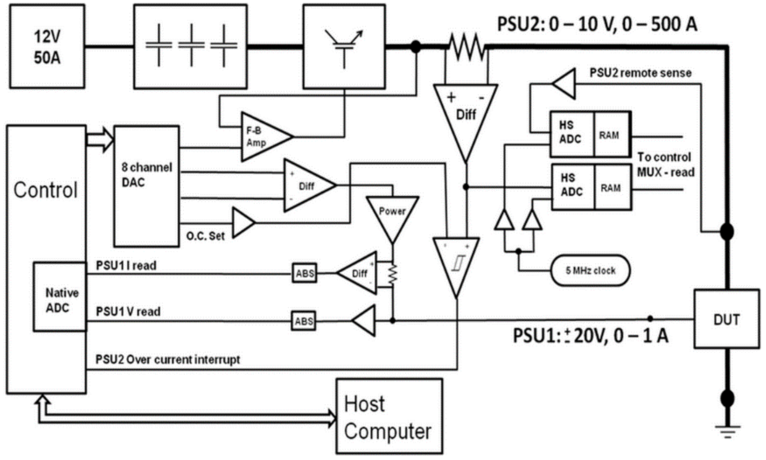
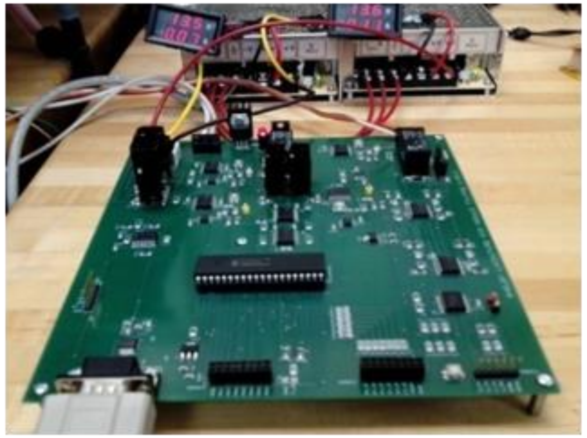
Engineered Electronic System --- Capability Gallery

High Current Device Characterizer

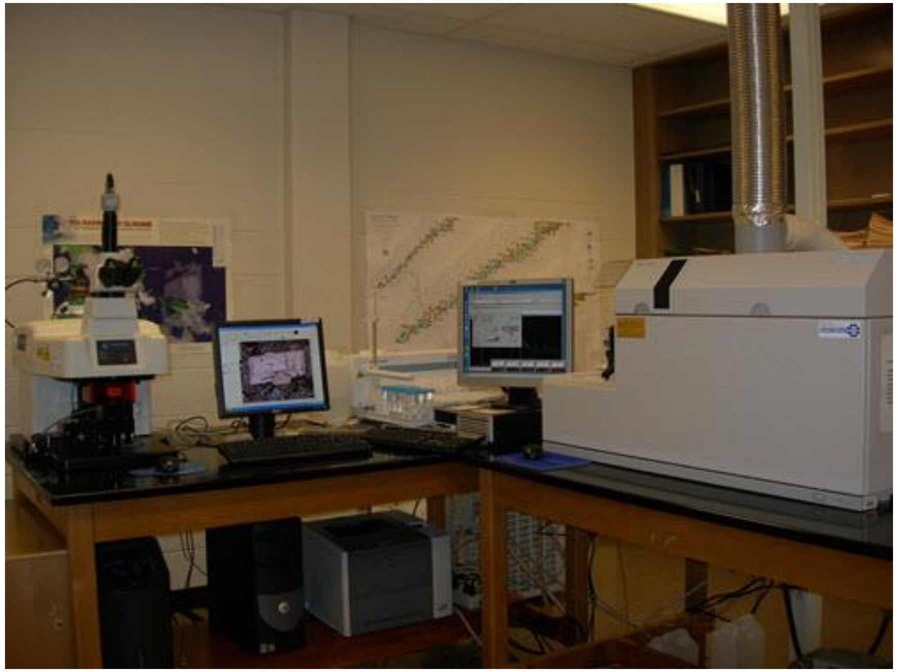


Narrow-Pulse Test Bed for Evaluation of Experimental Wide Bandgap Semiconductor Devices



Other Capabilities (partial list) See: https://www.depts.ttu.edu/phas/cees/Lab_Capabilities/

LA-ICP-MS (Geoscience Lab)



Agilent 7500cs inductively coupled plasma quadrupole mass spectrometer (ICP-MS) and a New Wave UP213 Nd:YAG laser system. The laser ablation sampling system can be used in conjunction with the ICP-MS to study trace element abundances in a wide range of solid materials.

CNC machining

Automated Orbital Welding

X-Ray scanning diffractometry (XRD) and XRF

Data Acquisition

- Data structures include temperature, pressure, flow rates, potentiometry, headspace mass spectra, ICR mass spectra, liquid ion chromatography, ambient O_2, ambient H_2, current, voltage, magnetic field, electric field, radiation (β, n, γ) counters and spectrometers, XRD / XRF, ED, BSD, BSED, EDS, imagery, etc.
  - Recorded on hard drives using National Instruments (NI) digitizers and / or TSIM continuous-capture file structures
  - Fast event data is automatically saved to a RAID drive when the signal variation exceeds 3σ over a designated time interval (~ 1 μs)

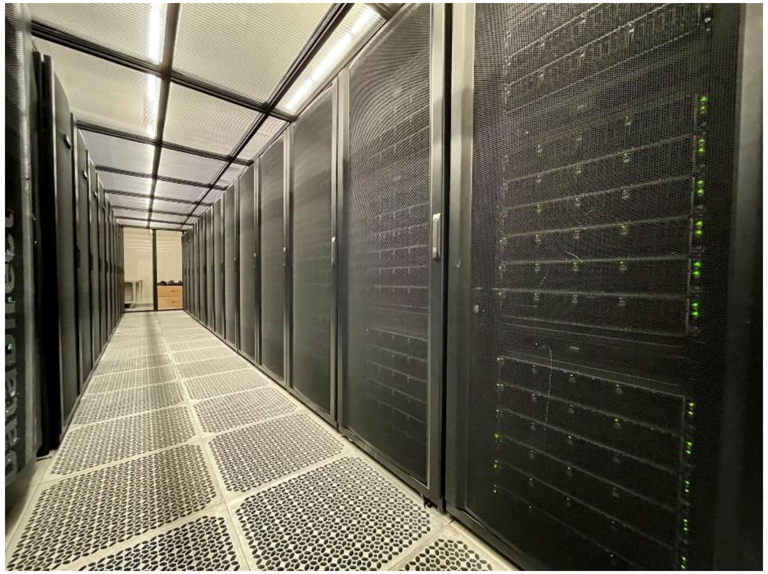
Measurement	Recording Method	Settings	Latency	Storage Media
NI (std) digitizers	Continuous	<= 50k sps	~ 10^{-8} s	Hard drive
Fast event	Data triggered	<= 50M sps	< 100 ps	RAID
Waveform Capture	2 GHz storage oscilloscope	~ 5G sps	~ 10 ps	Hard drive
EM spectrum	Analyzer w. RM	<= 110 GHz	Continuous	Hard drive

Modeling

- COMSOL Multi-physics (all modules), commercial license
- Nuclear Monte Carlo, N-Particle version 6.2
- Various custom lumped-element models, applied to thermal performance evaluations
- Codes run on work-stations, the TTU High-Performance Computing Cluster, and may code may be transported (if necessary) to Texas-wide computing assets, such as the Lone Star Cluster

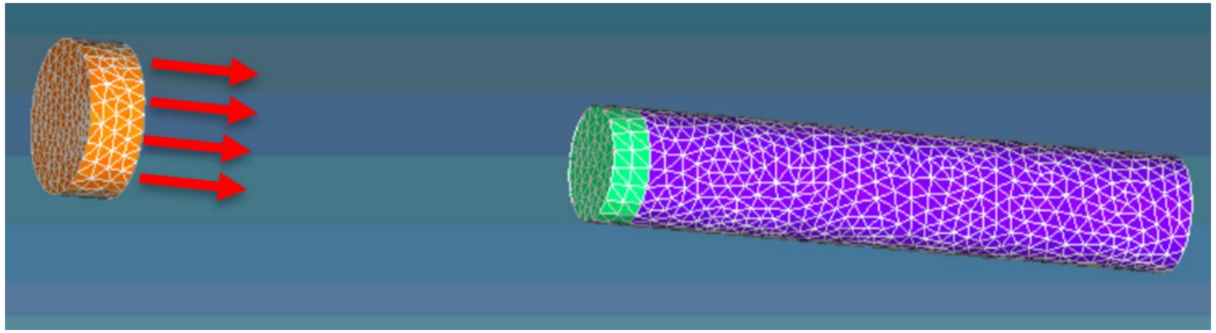
Modeling (Con't)

TTU HPCC

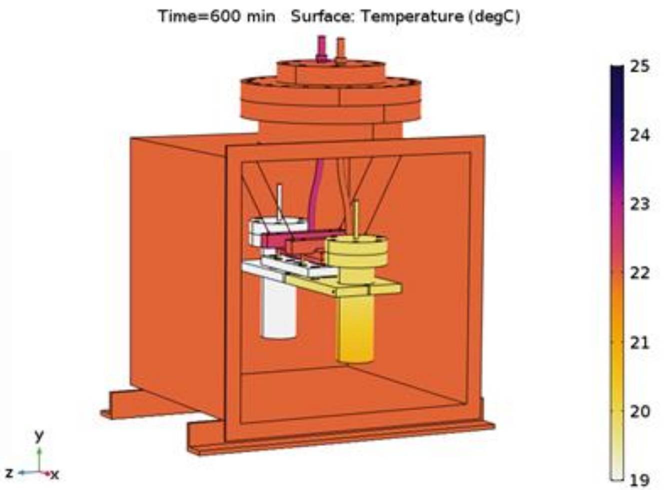


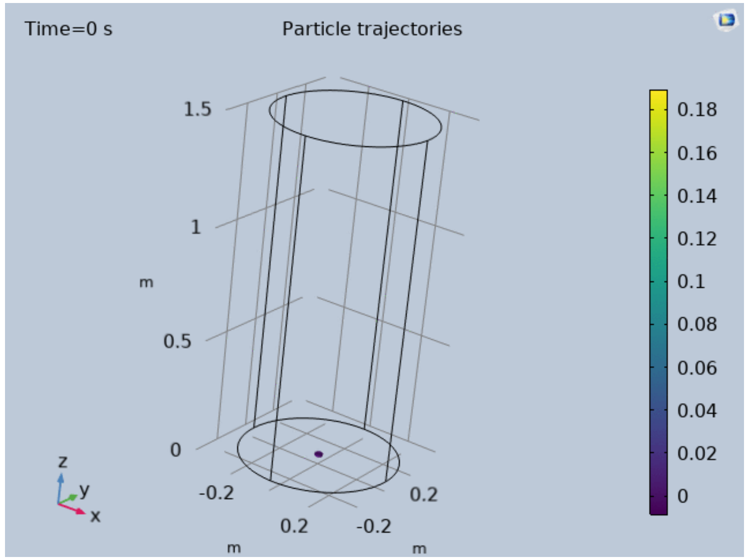
Partition:	Nocona	Quanah / XLQuanah	Matador	Toreador	Ivy
Type	CPU	CPU	GPU	GPU	Auxiliary CPU*
Total Nodes	240	467 / 16	20	11	50 / 2
Theoretical Max	983 TFLOPS	565 TFLOPS /19 TFLOPS	280 TFLOPS	287 TFLOPS	40 TFLOPS
Benchmarked	804 TFLOPS	485 TFLOPS / (N/A)	226 TFLOPS		N/A
OS	CentOS 8.1	CentOS 7.4 /CentOS 8.1	CentOS 8.1	CentOS 8.1	Rocky Linux 8.5 /CentOS 8.1

MCNP 6.2



COMSOL Multiphysics simulation





Initial Test Plan

- All PI Teams have been contacted to determine all service needs, including routine gas analysis, wide-area electrode mapping, and special design projects
- Special measurement support efforts identified to date include:
  - Development of a high-temperature (~ 500 ^{o}C) alpha particle detector for the Stanford team, which will be developed in collaboration with the other Cat. B team at U. Michigan, and other external collaborators
  - Methods to characterize stable element transmutation with the MIT Team, including:
    - Wide-area BSD, SED, and EDS mapping of electrodes
    - Develop ablative methods to expose and analyze sub-surface transmuted stable elements
  - Continuing discussion with PI Teams of other needs and of our abilities
    - Complete site visits during the week of September 18, 2023
- Legal work on Data Use Agreements is complete now, so we will proceed to negotiate with all PIs and put these plans in place.

Initial Results

- Milestone M1.1: *Refine Tasks and Milestones*, and M1.2: *Consultation on Experimental Design and Metrology*, are both 50% complete, and will be completed by the end of September, 2023. Consistent with SOPO plan when adjusted for dates when teams were placed on contract.
  - Support planning for the LBNL Team is set for an August 29 discussion.
  - Further support visits are planned for the week of September 18 with ETC, MIT Amphionic, and the Univ. of Michigan Cat. A Team (pending, subject to change)
  - Plans to support the other Cat. B Team as they support the high-temperature alpha detector design for the Stanford Team
  - Periodic team meeting are planned now with the MIT Team
  - Plans for gas sample capture has been completed with the Stanford Team, and with ETC
  - Work on wide-area mapping of the MIT Team's electrodes (following experimentation), using SE, BSE, and EDS sensors, is complete now and has been shared with the MIT Team
- Technical Data Agreement Draft complete and ready to negotiate with each Cat. A Team
- Vacuum Open-System Calorimeter draft submitted for publication
- All module designs complete for FT-ICR electronics replacement to I2C

Plans for Next Quarter, October – December, 2023

- Finalize Milestones M1.1 and M1.2 by the end of September, 2023
- Complete Milestone M1.3: *Complete upgrade plans for technical capabilities*, by December 15, 2023
- Complete Milestone M3.1: *Train new personnel,* by October 31, 2023. This is nearly complete now. Umer Farooq is fully trained in FT-ICR and will soon learn TEM techniques. Sandeep Puri has completed TTU's electron microscopy class, and will soon be fully trained in the Zeiss Crossbeam 540 SEM / FIB system
- Negotiate and sign Data Agreements with each Cat. A team by December 31, 2023, consistent with *Go / No-Go* Milestone M1.4. Extra Credit: Enter into a similar data agreement with the Cat. B Team by the same deadline
- Complete Milestone M4.1: *Upgrade legacy electronics and test upgraded prototypes for accuracy and stability*, by December 31, 2023
- Continue to deliver metrology support as agreed to all Cat. A teams