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Research Article

The Center to Study Anomalous Heat Effects [AHE] at Texas Tech University

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Abstract

The Center for Emerging Energy Sciences at Texas Tech University (CEES) has been established to explore critical parameters in the observation of the anomalous heat effects (AHE). A large number of experiments report the production of heat from metal samples loaded with hydrogen or deuterium in amounts that are often thousands of times greater than the enthalpies of possible chemical reactions. The effect is anomalous because there is no agreed-to mechanism, and particle radiation rates are not reported at levels that are consistent with any known nuclear process.

Keywords: Calorimetry, Cryogenic, Electrochemistry, Heat-helium, Spectroscopy

1. Basis of Work

Heat-helium correlation was first documented by Miles [1,2] and co-workers in 1991 stating convincing evidence that their measured excess enthalpy correlated with D+D $\rightarrow^4$He + 23.4 MeV fusion reaction (by some unknown, and presumably many-body process). These same results have been reported by at least seven laboratories in three countries (including two of the present authors in three different experimental modes at SRI and ENEA).

While necessary improvements have been made to calorimetry, triggering and data acquisition, much is still left to be understood about the process and mechanisms of excess heat release. The intent of the work described here is not to verify nuclear processes, specifically, but to gain a fundamental understanding of the mechanisms at play. Extensive

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materials characterization and eventual full reproducibility is the framework upon which the work performed at the AHE Center at Texas Tech. University (TTU) will be built upon, permitting direct correlation of excess heat production to materials properties that will point to the mechanism of this currently anomalous effect.

2. Strategy

Sufficient characterization of the metal foils both before and after their use in electrolytic cells is a critical path for correlation studies. While this characterization technique has yet to be standardized, the authors have embarked upon a path to define the necessary information to identify parameters that correlate with anomalous heat release.

Large scale, high resolution imaging is essential to investigate surface features of the cathodes. This will be achieved by use of atomic force microscope (AFM), scanning electron microscope (SEM), and confocal microscope (CM). All of these facilities are available at the Imaging Center at TTU which shares the same building as the AHE Center at Texas Tech.

To achieve nanometer resolution of areas $\sim 1 \times 4$ cm will require automated image acquisition and stitching software integrated with the SEM to register location. The registration will allow for the user to easily return to regions of interest for imaging at higher resolutions. Example foils included here were provided by the Violante Group at ENEA [3].

Contact with FEI regarding equipment upgrades for this project resulted in small demonstration in which the palladium foil was scanned and automatically stitched together as the scan progressed. The images below show resolution of this composite scan at several levels of magnification.

Additionally, plans to capture of the full spectrum of RF emission that have recently been observed to correlate with AHE production at TSEM, NRL, ENEA, and other laboratories is in progress. The effort described here will be led by the AHE Center at TTU and a collaborative agreement has been set in place with ENEA – Frascati (the Violante Group) to lead the further study of heat-helium correlations.

3. Electrolytic Cells

Based upon the pioneering work of Fleischmann and Pons, which has been further developed by McKubre [4], Violante [5], and other authors, correlation and rates of excess heat and helium production of deuterium and/or hydrogen loaded palladium foils will be investigated. While this work will continue at the established ENEA facility, the AHE Center at Texas Tech will establish working cells to begin testing characterization techniques, described in *Strategy,* in the next few months.

When satisfactory operation is achieved at Frascati, closely similar apparatus will be assembled in the AHE Center at TTU to check site-to-site repeatability. It is possible that a third laboratory will be selected in the future to further check repeatability.

We have purchased a PXIe system from National Instruments to aid in the data acquisition and processing necessary for calorimetric results. The system is equipped with an Intel Core i7, 12-bit oscilloscope/digitizers, DMMs, 26.5 GHz spectrum analyzer, simultaneous data acquisition (ADC/DAC + DIO + Timing/Triggering), and 5.7 TB RAID drive.

4. Exploding Wires

Considerable effort has been made to decrease data acquisition times, and therefore experimental setup and turnover times. As such, deuterium loading of palladium wires has emerged as a feasible alternative to foils. However, the process of loading the wires requires ultra-pure reagents in an ultra-clean environment, which introduces a different set of experimental requirements.

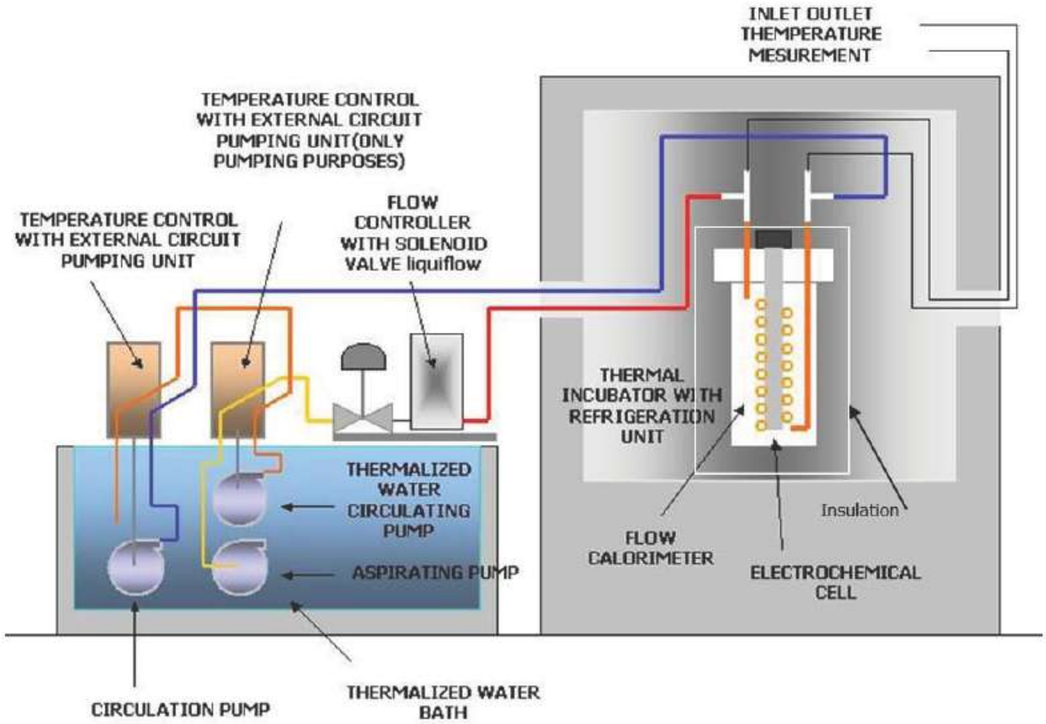


Figure 1. ENEA mass flow calorimeter.

The image below summarizes initial efforts made by Tanzella, Bao, and McKubre [6,7] to examine electrolytically loaded wires of various composition. The area below the line represents a region of no anomalous heat release, points above the line exhibit a significant percentage of excess heat. The AHE Center at TTU will be exploring the parameter space developed here in pursuit of correlations between the extent of heat release and helium production. Once operation is reached, site-to-site repeatability will be checked at Frascati.

5. Preliminary Results

During the last three months, we have investigated the necessary components for sufficient optical characterization of the electrolytic foils. Currently we have use of a Hitachi S4300 SEM which can be upgraded to provide surface feature resolution at the nanometer-scale, Energy Dispersive X-ray Spectroscopy (EDS) results for compositional information, and Forward Scatter Detector (FSD) imaging to detail the crystal structure orientation of the entire surface of each foil. Examples are shown below of the result of 33 stitched individual images with 30% overlap to guarantee feature matching. No smoothing has been applied.

Image defines sample as pure palladium with no impurities. Colors are associated with spectral markers in SEM image above. Red: La - K_α. The sample above shows a very strong preferred orientation with the $\langle 001 \rangle$ lattice orientation at the foil's surface.

Following preliminary tests with comparable models at Hitachi, EDAX, Oxford, FEI and Zeiss, TTU is in the process of installing a new Zeiss XB540 Crossbeam Focused Ion Beam FE SEM with Secondary Ion Mass Spectrometry

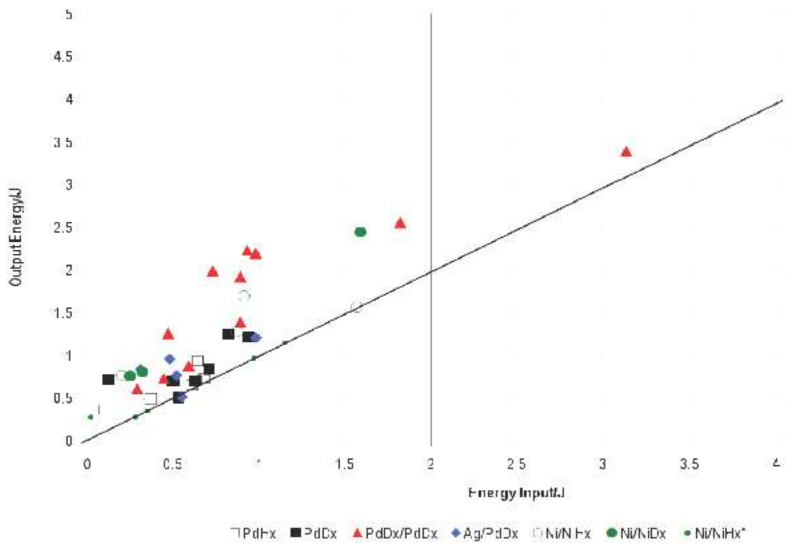


Figure 2. Calorimetrically determined output energy versus Joule input energy for a range of electrochemically loaded wires exercised electrically to destruction.

depth profiling (FIB-SIMS), material contrast with simultaneous Inlens SE and ESB imaging and Atlas 5 designed to create comprehensive multi-scale, multi-modal images with sample-centric correlative environment. The software capabilities include correlation of X-ray microscopy and FIB-SEM data to localize sub-surface features in 3D to target FIB sites that are not visible on the sample surface.

6. Mass Spectrometry

The AHE Center at TTU is in the process of establishing three separate methods of low-AMU mass detection for use in the previously mentioned experimental designs:

(1) *Leak Detection*. The Pfeiffer ASM 340 leak detector is calibrated for detection of H_2, ^{3}He, and ^{4}He, with

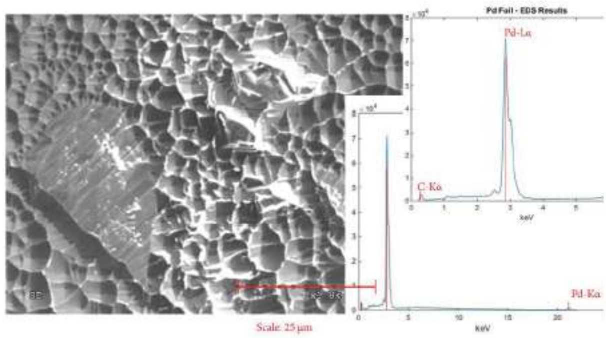


Figure 3. SEM and EDS results of a pure palladium electrode using Hitachi S4300 at TTU Imaging Center.

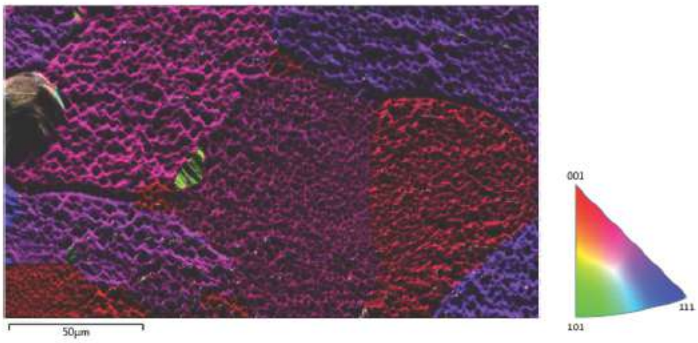


Figure 4. FSD and Euler Color Map overlay of palladium foil seen in Fig. 3 using the same Hitachi S4300 instrument.

a minimum detection rate of $< 5 \times 10^{-12}$ Torr l/s. This apparatus will be used to ensure that all seals are hermetic, but can also be utilized as a baseline calibration for background levels of the species listed above.

(2) *Gas Chomatography – Mass Spectrometer (GC-MS).* The JOEL GCMate is a compact double focusing mass spectrometer that incorporates a gas chromatograph before a traditional mass spectrometer that has a range that extends as low as m/z 1–3000. The figure to the right was obtained from JEOL by monitoring H_2^+ and D^+ from a mixture of D_2O and H_2O (mass difference of 0.00155 amu) with a resolving power of 3400. This instrument matches that used at ENEA – Frascati to facilitate in experimental repeatability once the AHE Center at TTU receives the mass flow calorimeter as described above.

(3) *Fourier Transform Ion Cyclotron Resonance (FT-ICR).* The Quantra Advance Mass Spectrometer utilizes a 1 Tesla magnet field ideal for confining small masses and lower voltages for small mass ion control. The piezo-electrically driven injection valve controls size and duration of sample sizes down to picoliter volumes while maintaining a consistent 10^{-10} Torr base pressure. Currently equipped with a 5 MHz waveform generator, the lowest detectable mass is 5.6 amu with a resolution of approximately 10,000, as it was originally designed for masses 2–1000, but utilizing the second harmonic helium and hydrogen can be trapped. Upgrading to a 40

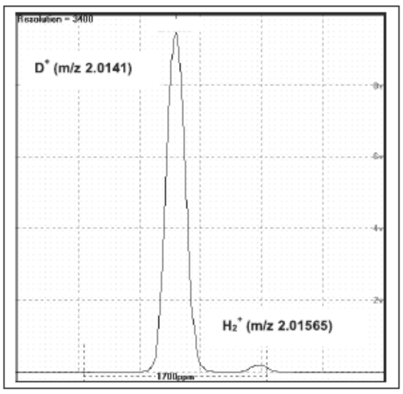
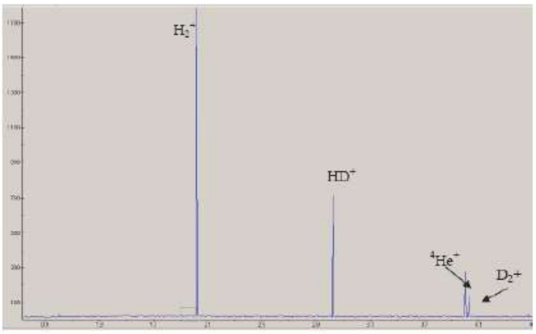


Figure 5. Illustrating the resolving power of the JEOL GC-MS, MS-071000A brochure available online.



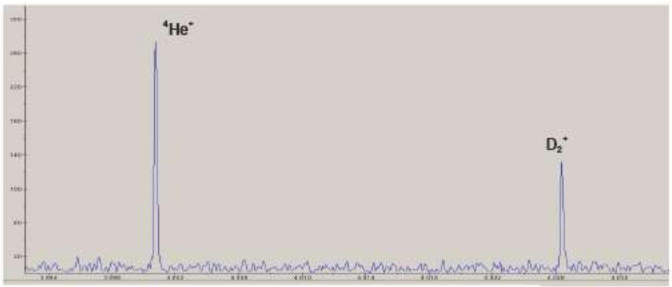


Figure 6. Plots of low mass spectrometer resolution provided by Siemens.

MHz generator would move detection limit to masses greater than 0.7 amu with a resolution of 400,000 for hydrogen and 200,000 for helium.

7. Custom Cryogenic Design

To initiate work on the exploding wire variations, Janis Research has assisted in the development of a custom research cryostat designed to operate with liquid argon (87 K), liquid nitrogen (77 K), or liquid helium (4 K).

The expected consumption rate of LAr is ~ 0.4 L/h with radiation baffles and bubble diffusers to ensure thermal stability of boil-off measurements before and during experimentation. Neutron and gamma ray transparent windows have been included in line with wires for emission detection with AmpTek X-123 Charge Drift Detectors.

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