

UNLV Research Foundation High Temperature Heat Exchanger Development

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Manager

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PDP 22

Overview

Timeline

- Project start date: 9/03
- Project end date: 9/08
- Percent complete: 50%

Budget

- Total project funding
 - DOE/NE: \$5,700k
- Funding received in FY05: \$1,930k
- Funding for FY06: \$1,870k

Barriers

- Barriers addressed
 - Nuclear Hydrogen Initiative R&D Plan – Material performance and component design and testing for: intermediate heat exchanger and high-temperature thermochemical water splitting (H_2SO_4 decomposition and HI decomposition). Improved materials for High Temperature Electrolysis.

Partners

- UNLV, UC Berkeley, MIT, General Atomics, Ceramatec, Argonne National Lab

Objectives

To assist DOE-NE in the development of hydrogen production from nuclear energy through:

- Identification and testing of candidate materials for heat exchanger components.
- Design of critical components in the interface and sulfur iodine thermochemical process.
- Fabrication and testing of prototypical components.
- Innovative materials development.

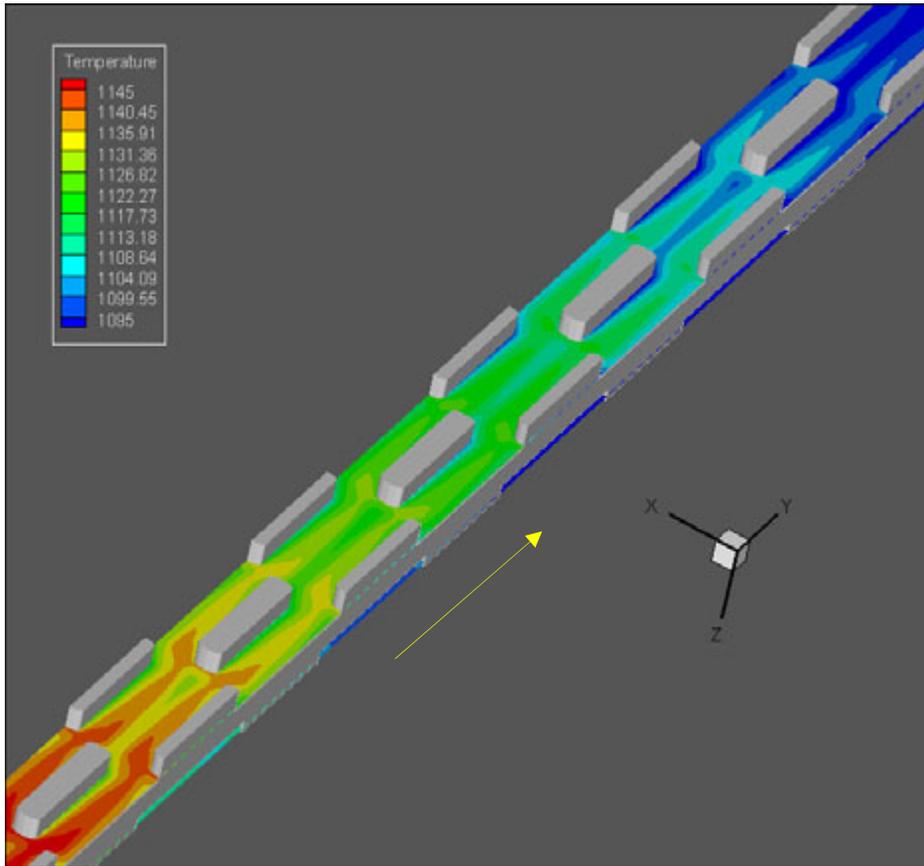
Approach

- Task 1: Heat Exchanger Component Design
 - Optimization, transient hydrodynamic, and thermal studies of off-set strip fin HTHX
 - Numerical analyses with chemical reactions and optimization studies for Ceramtec sulfuric acid decomposer
 - Stress analysis and optimization of Ceramtec HTHX
 - Numerical analysis of metallic Heatric-type sulfuric acid decomposer
- Task 2: Identification and testing of candidate metallic materials for heat exchanger components
 - Evaluation of material strength and corrosion properties
 - Materials tested: Alloy C-22, C-276, Waspaloy, 617, and 800H up to 1000 C.
 - Nb-1Zr, Nb-7.5Ta, Zr705, Ta-2.5W, and Ta-10W up to 400 C (for HI decomposition).
- Task 3: Heat Exchanger Prototype Testing
 - Experiments with surrogate materials to validate hydrodynamic and overall heat transfer coefficients from CFD results
 - Prototype to model ratio is 1:3 for the off-set strip fin design
- Task 4: Analytical Studies of the Effects of Acid Exposure on Structure Materials
 - Elemental analysis and bonding structure to identify phenomena (e.g. oxide formation and thickness)
- Task 5: Efficiency Improvement and Cost Reduction of Solid Oxide Electrolysis Cells
 - Use of dense film electrodes and electrolyte made by Atomic Layer Deposition
 - Elucidate the surface and bulk reaction mechanisms at the O₂ electrode
 - Determine the oxidation state of the nickel and the structure of the electrode/electrolyte interface during cell operation

Approach

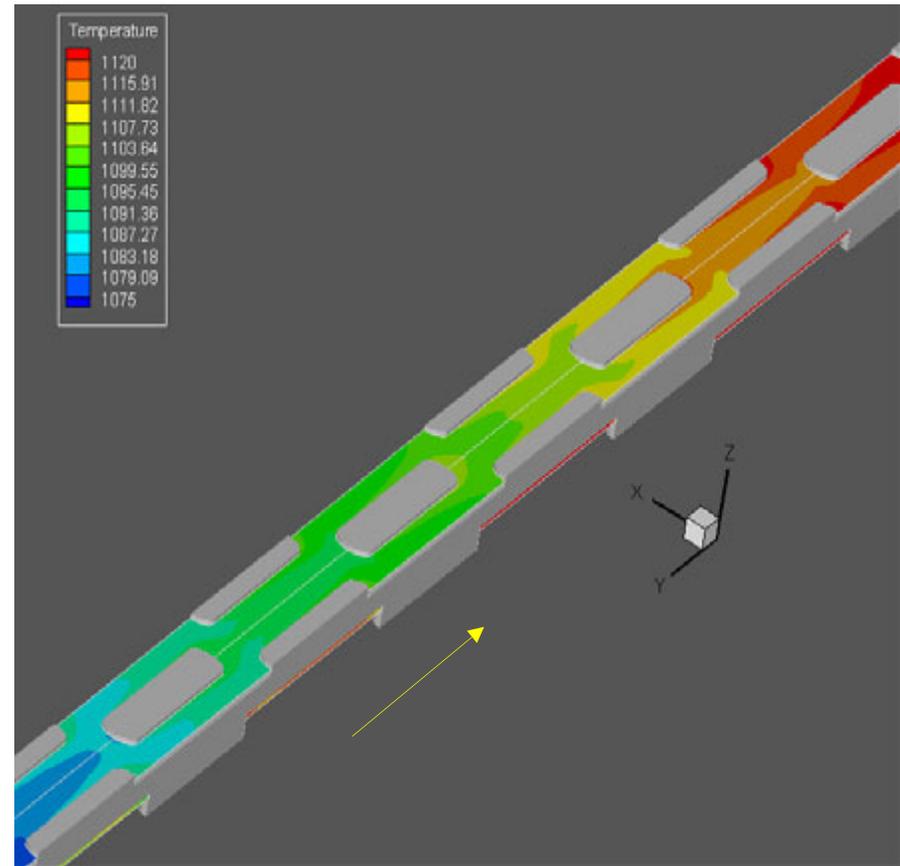
- Task 6: Corrosion and Crack Growth Studies of Materials in Hlx Environment
 - Screening of 22 candidate materials using immersion coupon exposure
 - Long term testing including crack initiation and growth studies and cladding options
- Task 7: Ceramic-Based High Temperature Heat Exchanger Development
 - Evaluation of mechanical and thermal properties of preferred ceramic materials
 - Heat exchanger design. Numerical modeling of performance, durability and fabrication economics.
 - Validation through prototype fabrication and empirical testing.
- Task 8: Materials Design and Modeling for C/SiC Compact Ceramic Heat Exchangers
 - Demonstration of C/SiC composite heat exchanger fabrication using polymer infiltration and pyrolysis (PIP).
 - Perform C/SiC HX thermal and mechanical analysis
 - Perform comprehensive C/SiC heat exchanger safety analysis
- Task 9: Development of Self Catalytic Materials for Thermo-chemical Water Splitting Using the Sulfur-Iodine Process
 - Material chemistry identification, alloy procurement and metallurgical characterization
 - Determination of Catalyst Effectiveness
 - Determination of Mechanical Properties
 - Prototypic Shape Fabrication & Testing

Temperature (K) Contours in the Middle Region of an off-set strip fin heat exchanger



Helium side

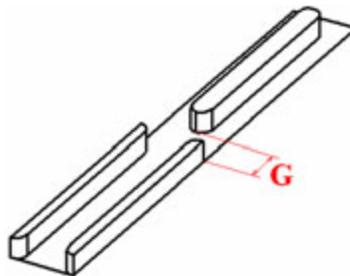
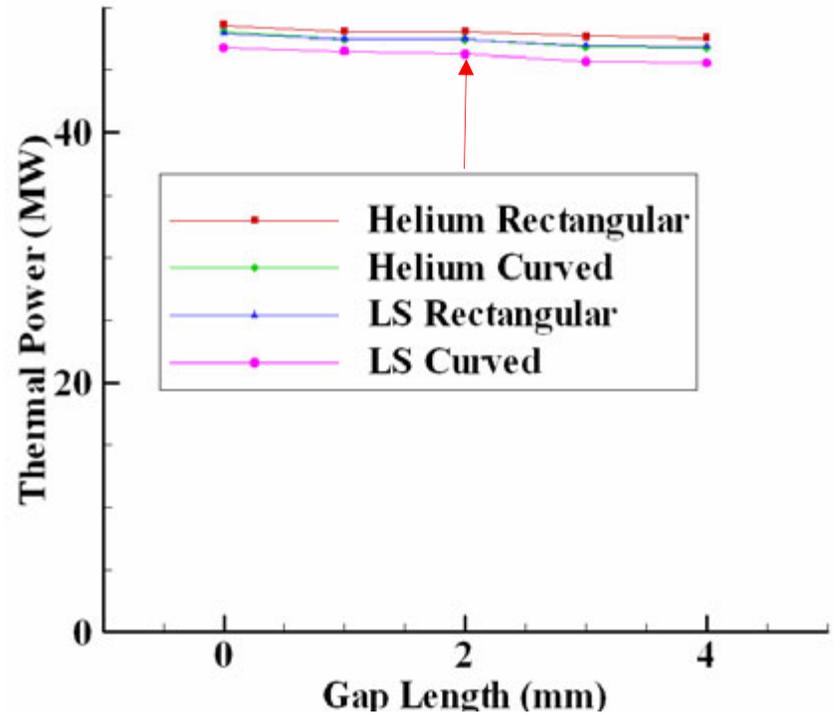
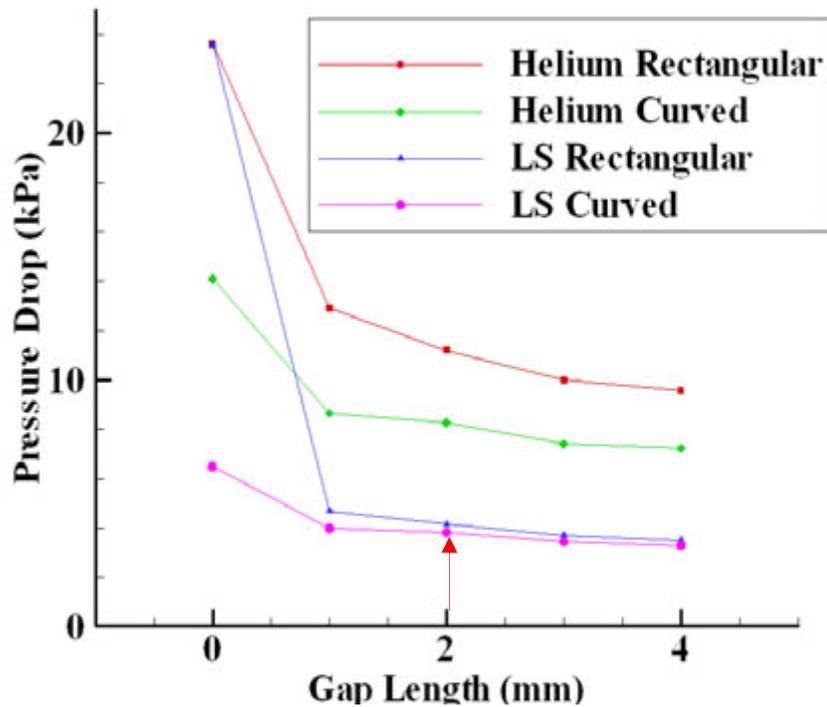
$$\Delta T = 377K$$



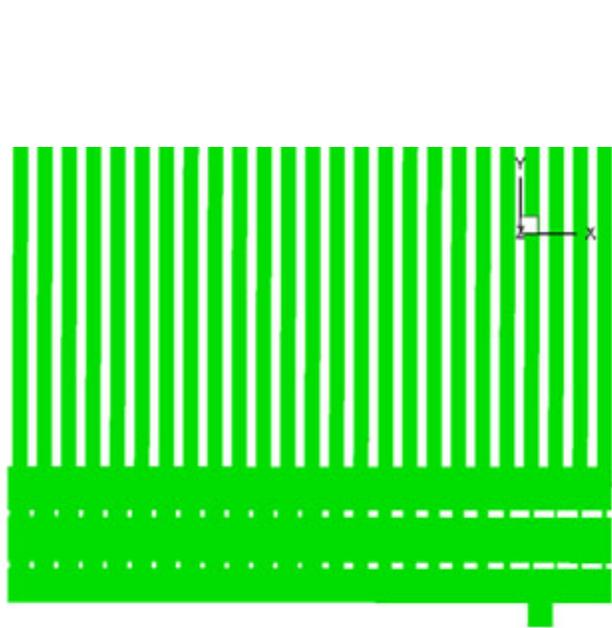
Liquid salt (LS) side

$$\Delta T = 419K$$

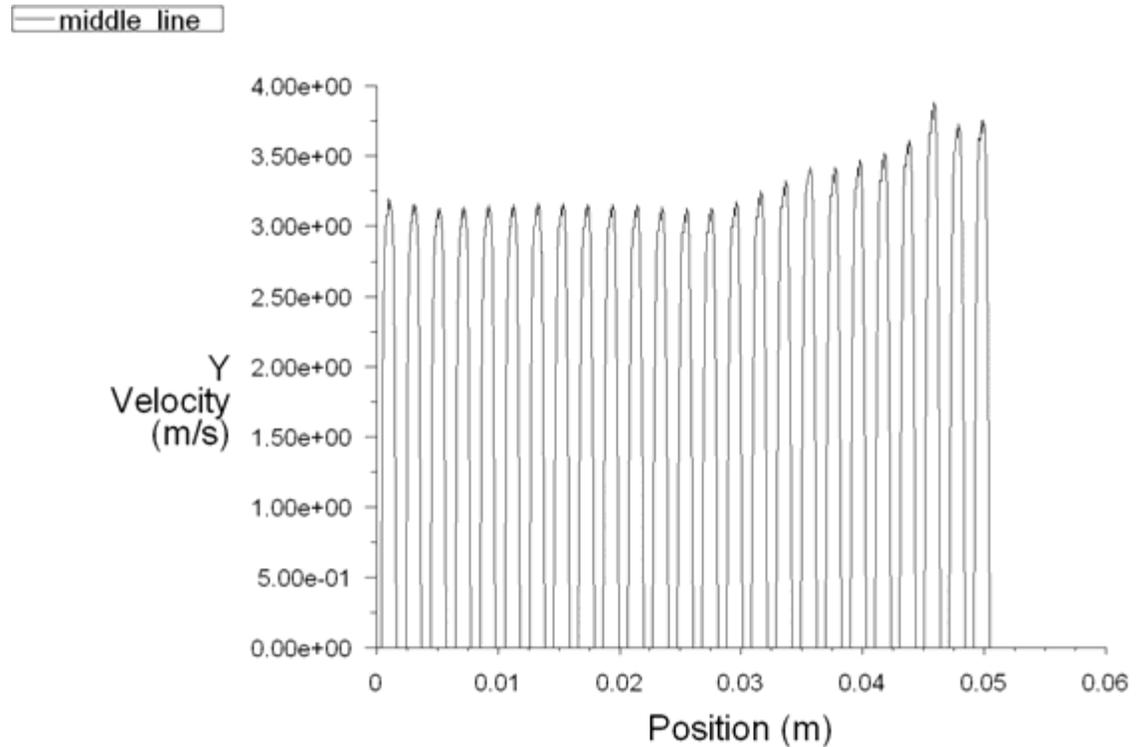
Effects of Gap Length on HX Performance



Velocity Distribution for the Optimized Geometry for the Ceramatec Sulfuric Acid Decomposer



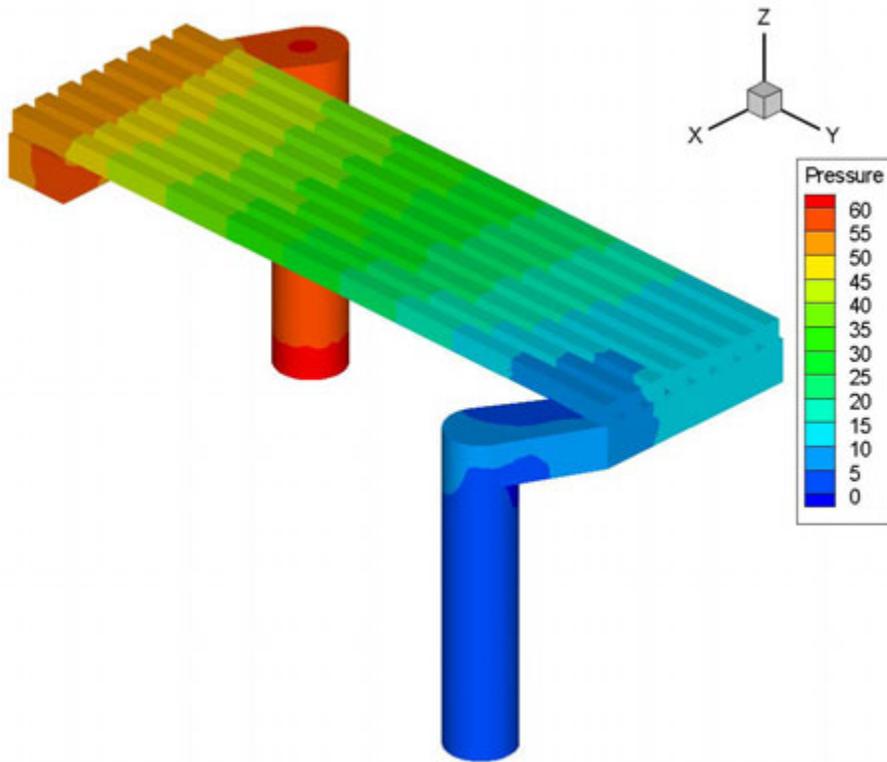
Optimized geometry



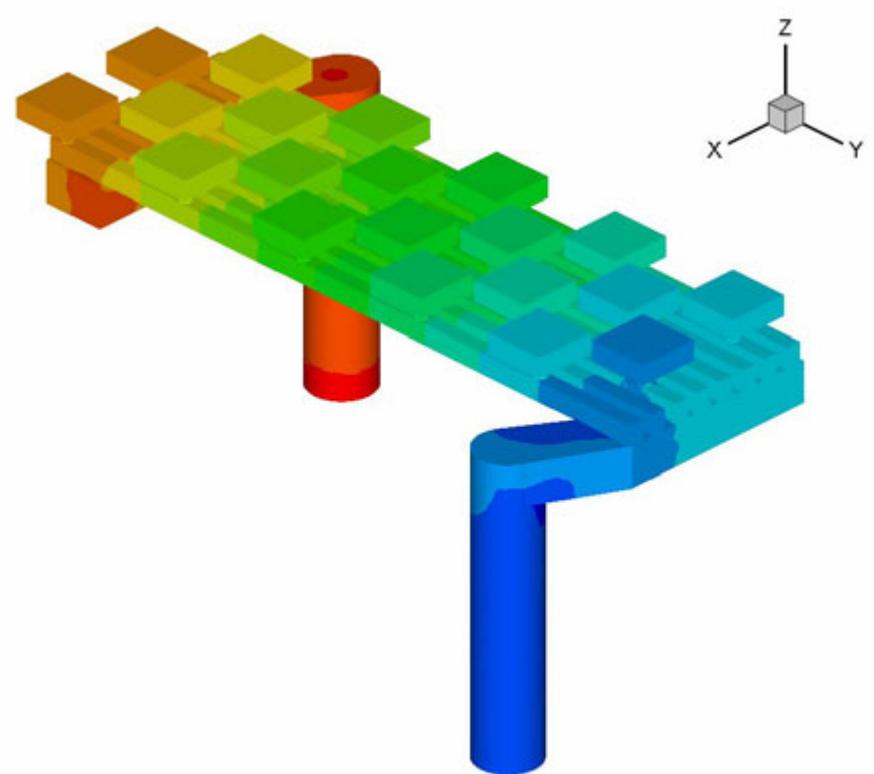
Y Velocity Aug 25, 2005
FLUENT 6.2 (3d, segregated, lam)

Velocity distribution at the midsection of the channels

Computed Pressure Distributions (Pa) for the Ceramatec Coupon Test



With no measurement channels

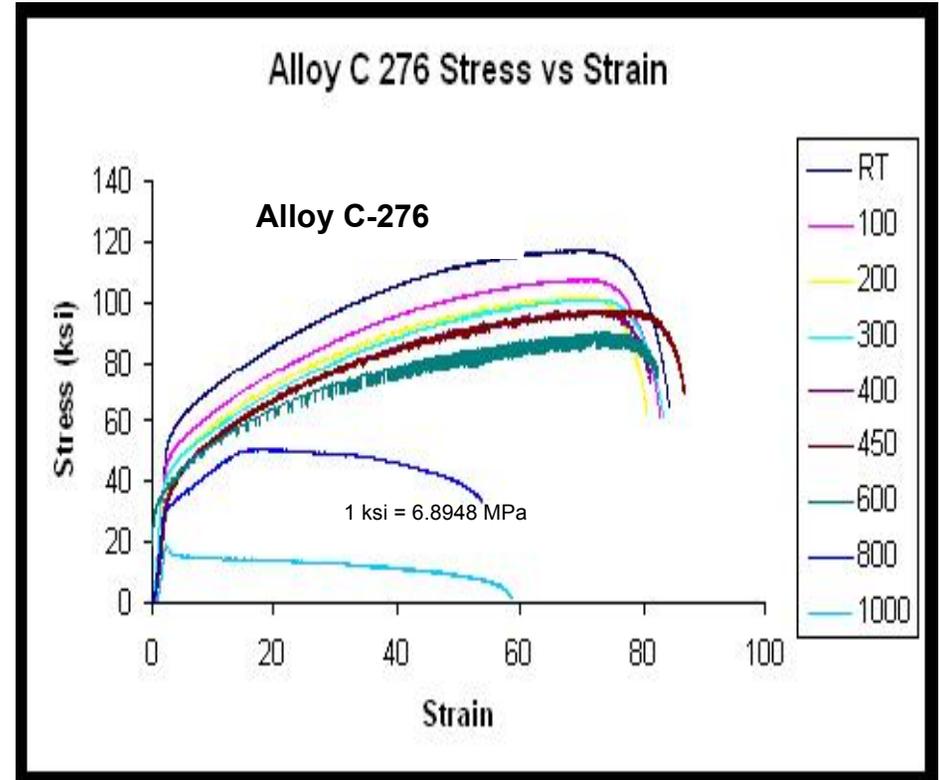
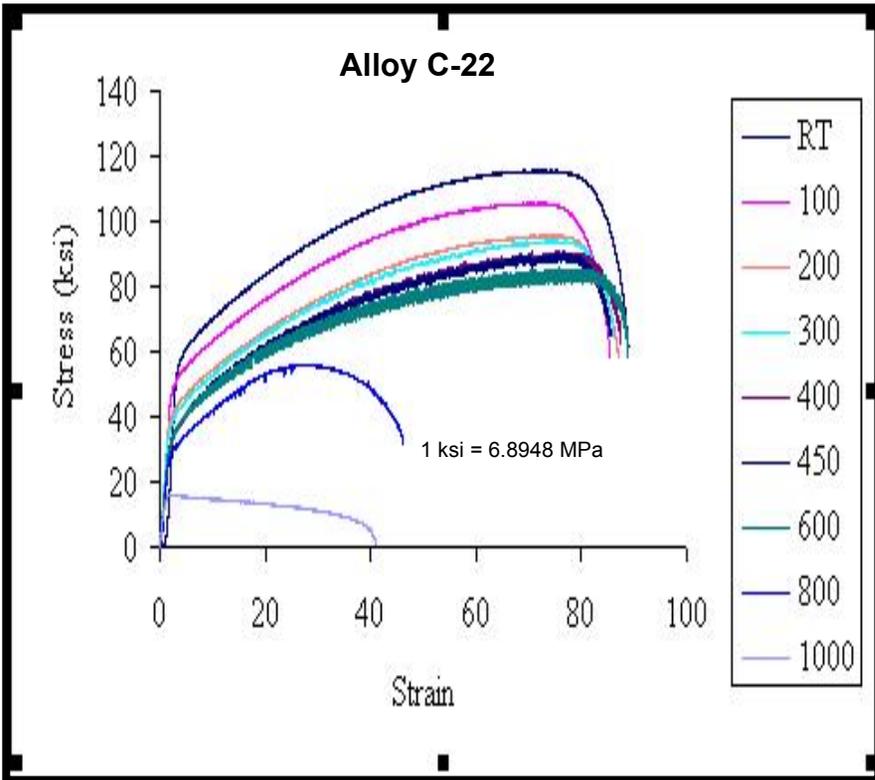


With measurement channels

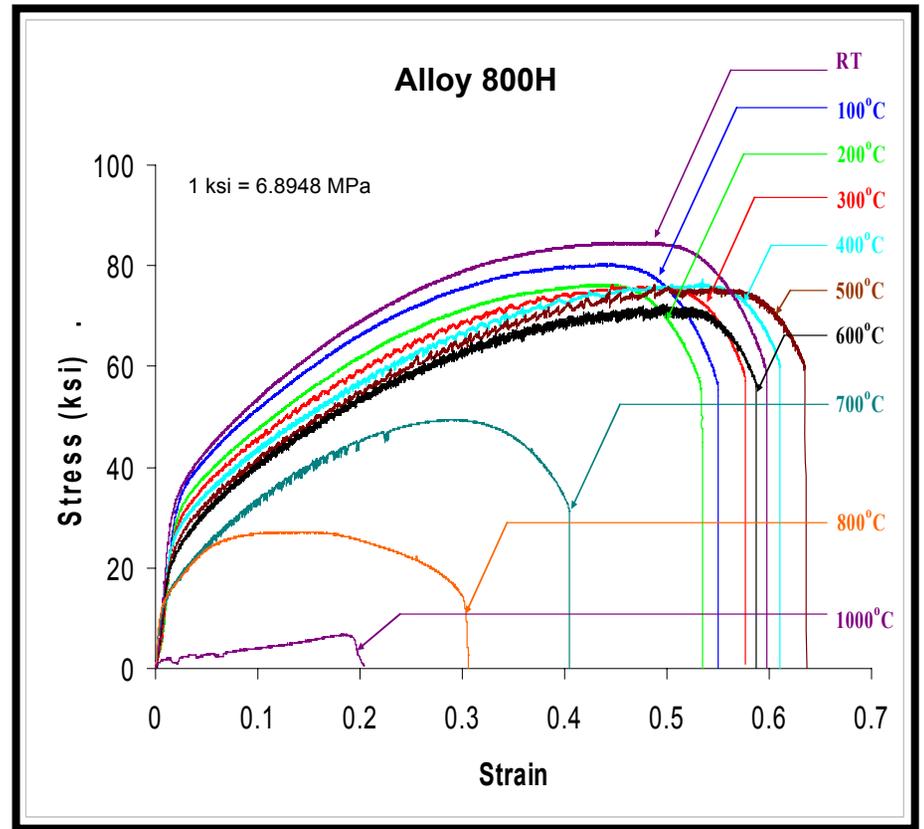
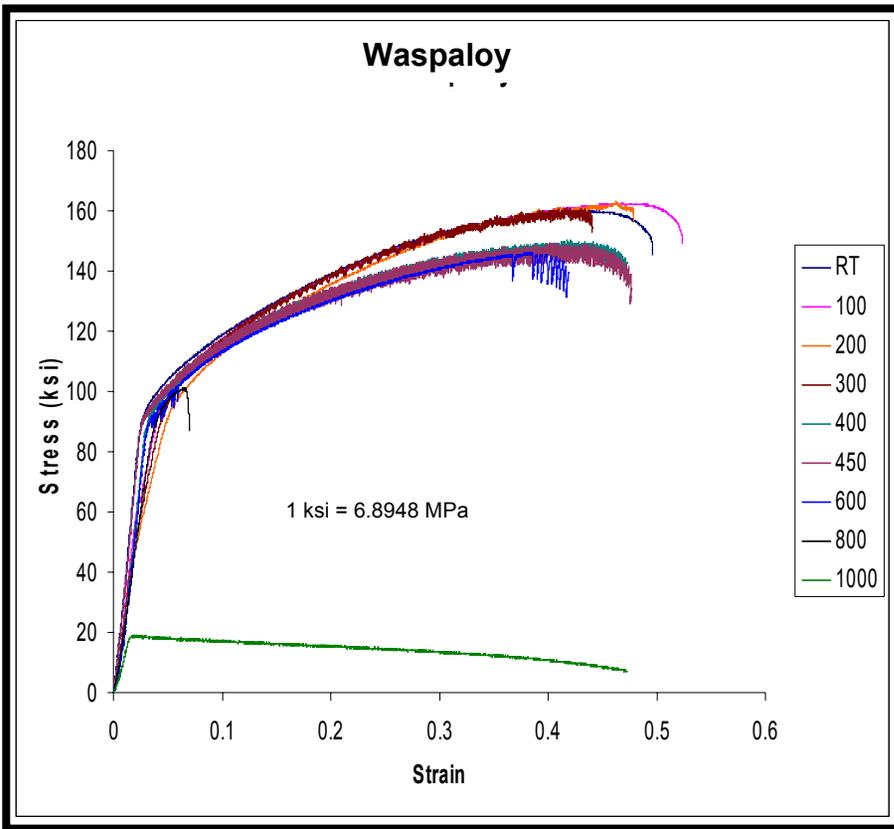
Accomplishments: Task 2: Identification and testing of candidate metallic materials for heat exchanger components

- Four structural materials, namely Alloy C-22, Alloy C-276, Alloy 800H and Waspaloy have been identified and characterized for heat exchanger applications.
- The results of tensile testing up to a temperature of 1000°C revealed a gradual reduction in tensile strength with increasing temperature, as expected.
- All four alloys exhibited significant ductility up to 600°C. However a significant drop in ductility was observed above this temperature.
- Failure strain for all four alloys were gradually reduced in a critical temperature range followed by its enhancement beyond this range. This phenomenon may be the result of dynamic strain ageing. For Waspaloy, a maximum dislocation density (ρ) was observed at 300°C
- Ductile failures were observed up to a temperature of 600°C for all tested materials. However, intergranular failure was observed with Waspaloy at 800°C possibly due to the precipitation of Boron at the grain boundaries.

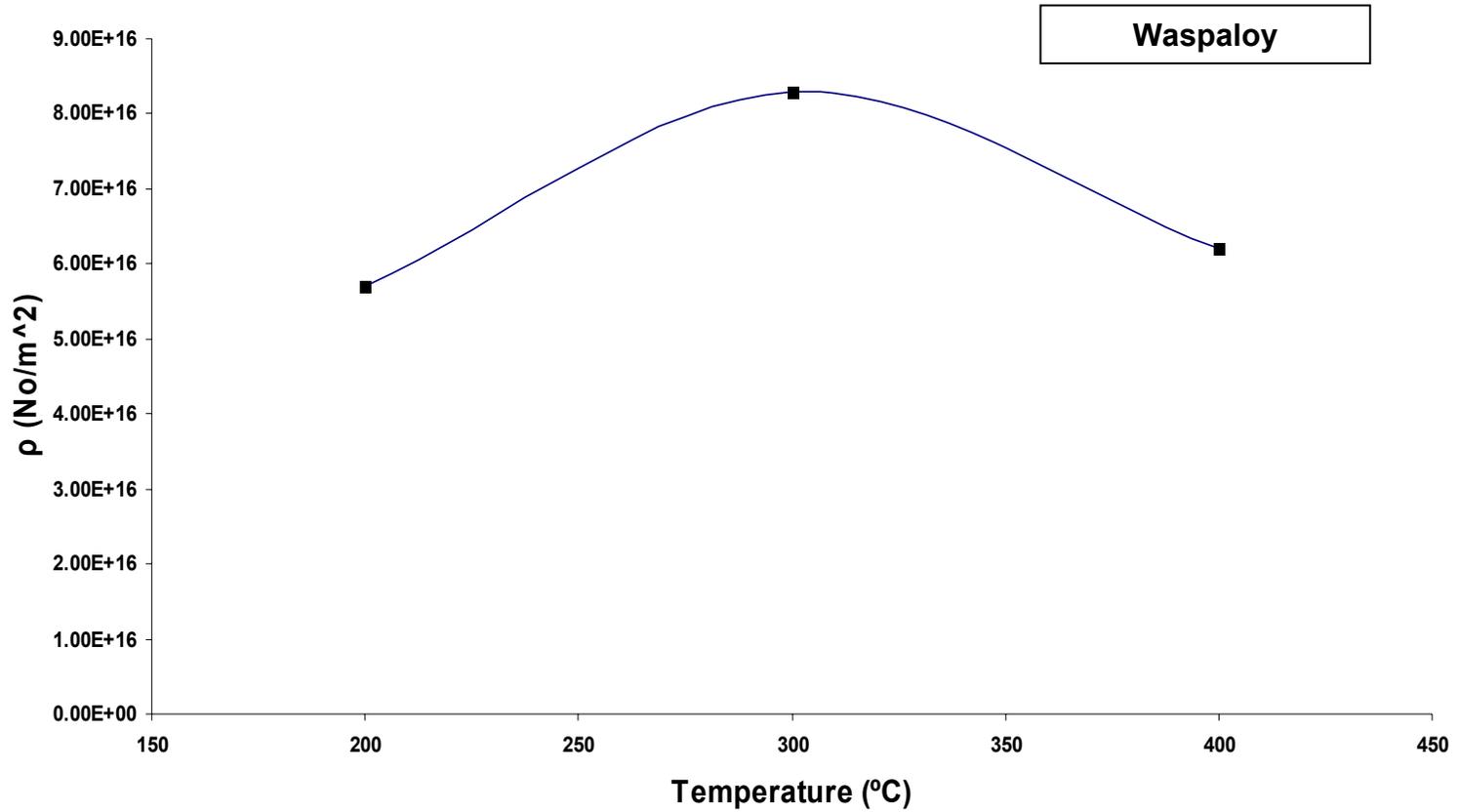
Engineering Stress vs Strain Diagrams at Different Temperatures



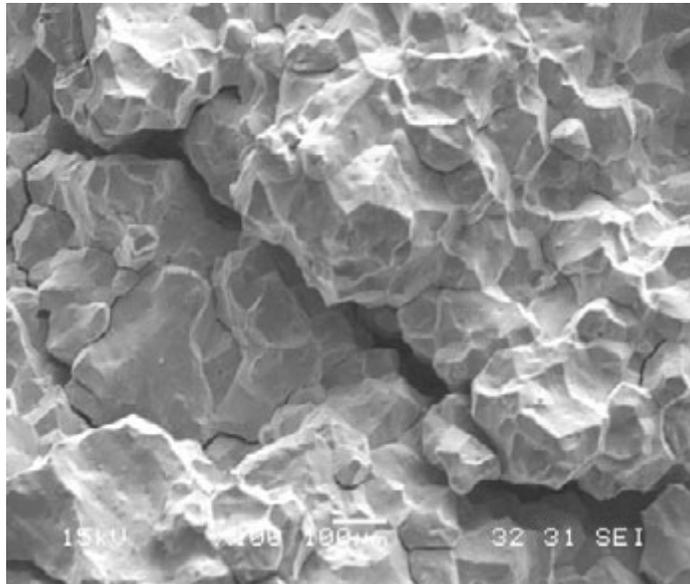
Engineering Stress vs Strain Diagrams at Different Temperatures



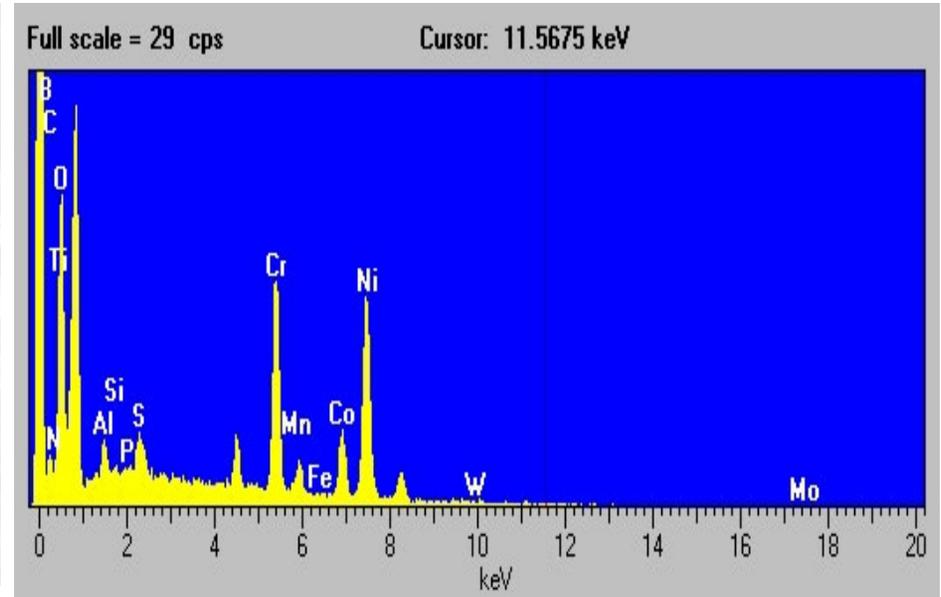
Dislocation Density (ρ) vs Temperature



Scanning Electron Micrographs



Waspaloy, 800°C, 100X

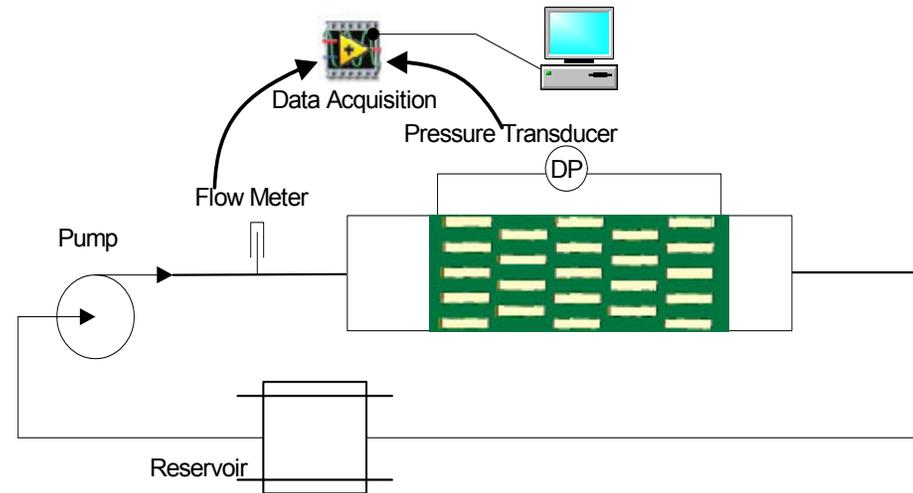
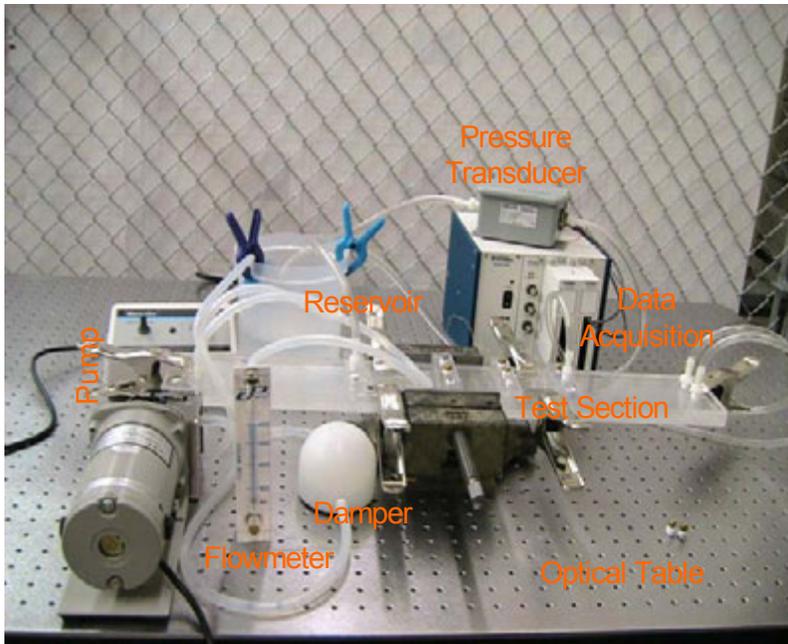


EDS Analysis of Waspaloy

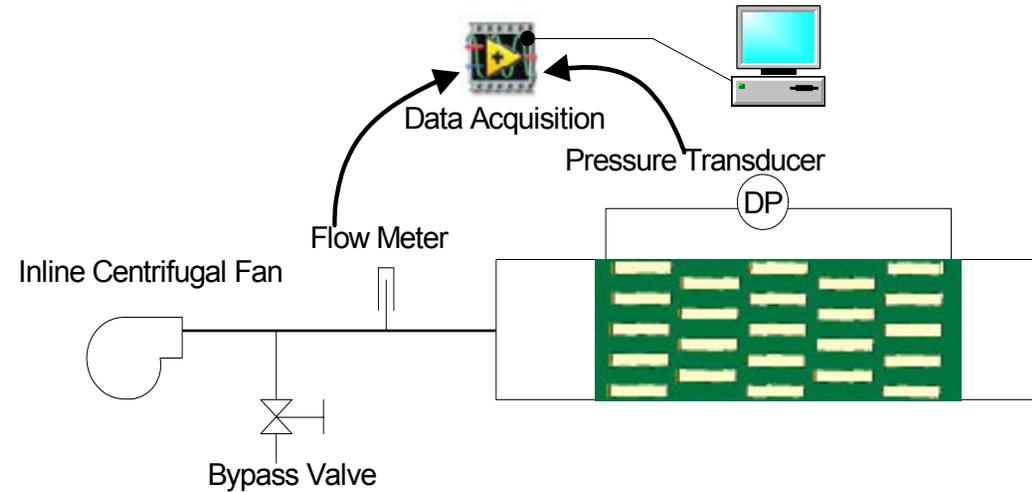
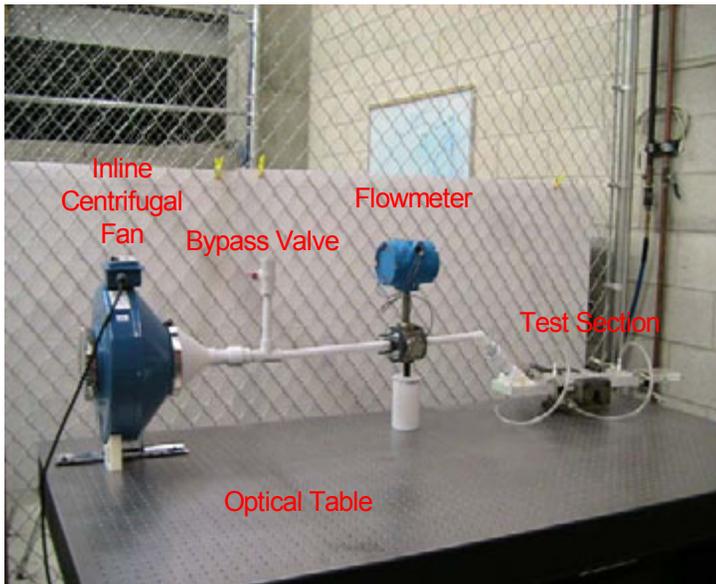
Accomplishments: Task 3: Heat Exchanger Prototype Testing

- Experimental testing lab is basically setup.
- 80% of the instrumentation has been ordered, installed, and tested.
- Hydrodynamic testing rig for a single-chamber test section with air as the working fluid has been setup and tested. Hydrodynamic tests began.
- Several problems associated with de-ionized water as working fluid in acrylic test section were solved: bubbles on the testing surface and sealing of the fins and walls.

Single-chamber Testing Setup with De-ionized Water



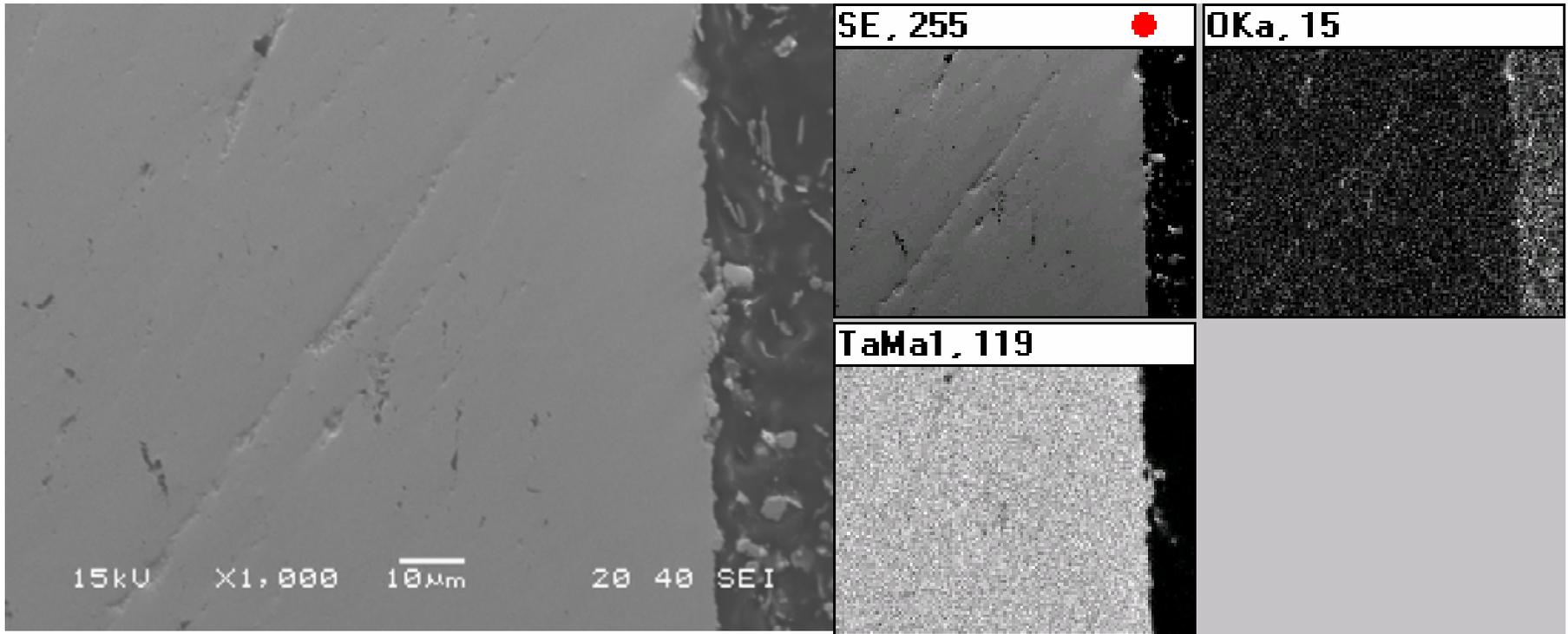
Single-chamber Testing Setup with Room Air



Accomplishments: Task 4: Analytical Studies of the Effects of Acid Exposure on Structure Materials

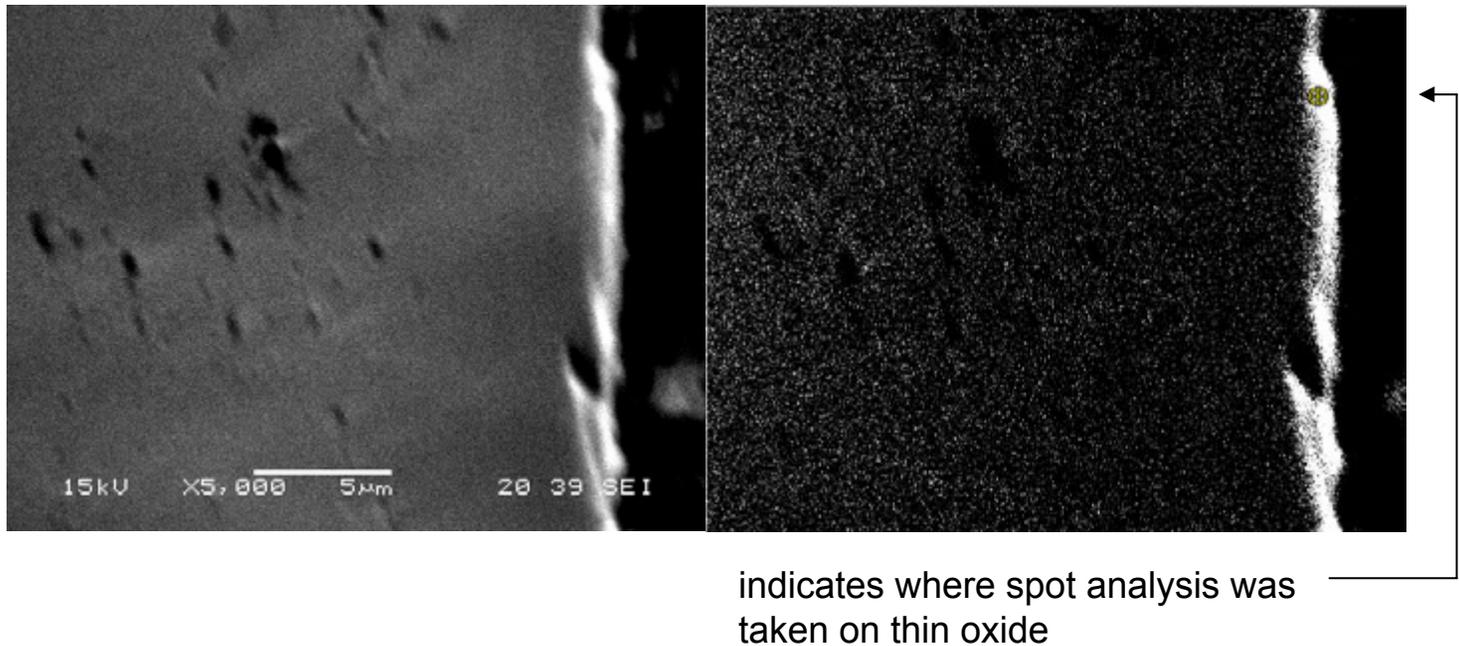
- SEM and XPS investigations performed on samples exposed to Hydrogen Iodide
- Oxide layers were characterized for Ta-2.5W, Nb-10Hf, and Nb-1Zr.
- Failure analysis was conducted for Nb-1Zr.

Ta-2.5W characterization



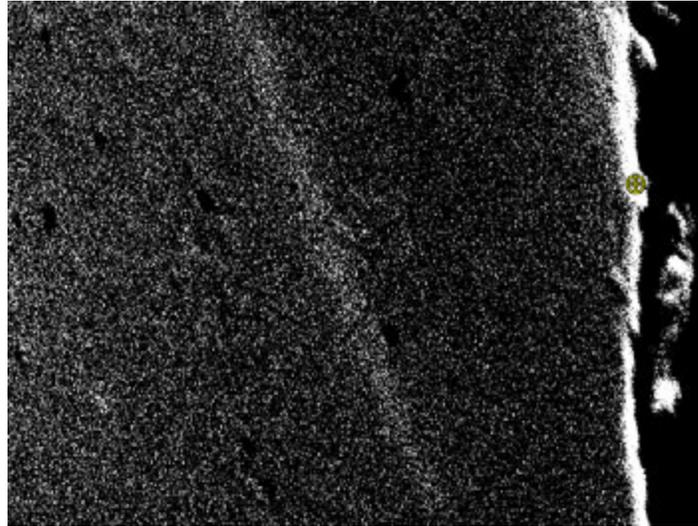
TaW – 1000 hours - Weld A side 1

Nb-10Hf Characterization



A 1-2 μ surface oxide, Nb₂O₅ by XPS was observed.

Nb1Zr Characterization



Nb-1Zr (unfailed) XPS showed an oxide layer that was of varying thickness from 200-1000 nm deep, composed of Nb_2O_5 . However, even at very shallow distances ($\sim 10\text{nm}$) significant reduced Nb was observed.

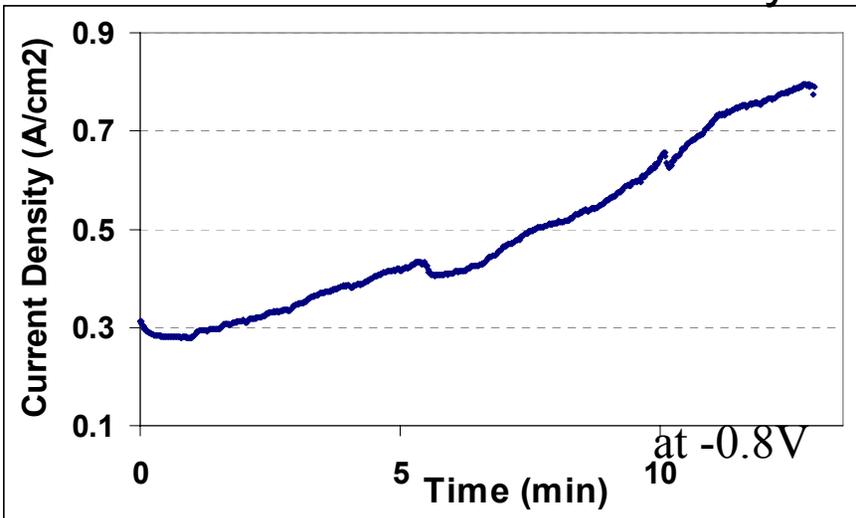
Accomplishments: Task 5: Efficiency Improvement and Cost Reduction of Solid Oxide Electrolysis Cells

- X-ray absorption and emission spectroscopy of reference powder materials of NiO, LaSrMnO, and ZrYO were performed.
- Synchrotron experiments at the Advanced Photon Source (ANL) were conducted in February and March 2006, films will be sent to UNLV.

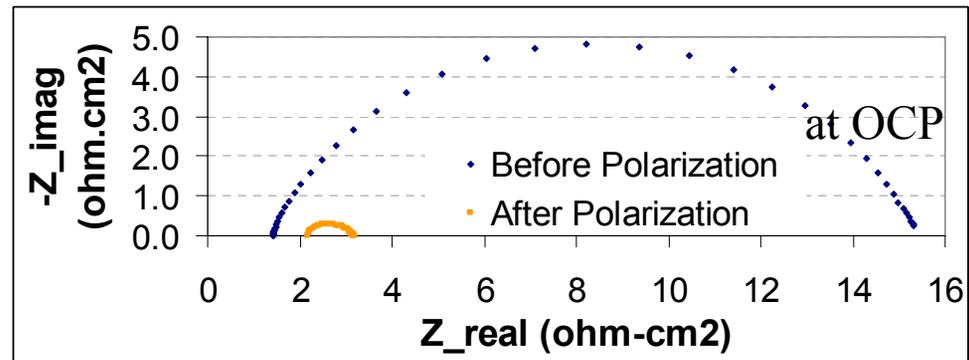
Electrochemical Characterization

Results for the 150 nm reference LCM electrode at 800°C

Potentiostatic measurements
for the electrode current density

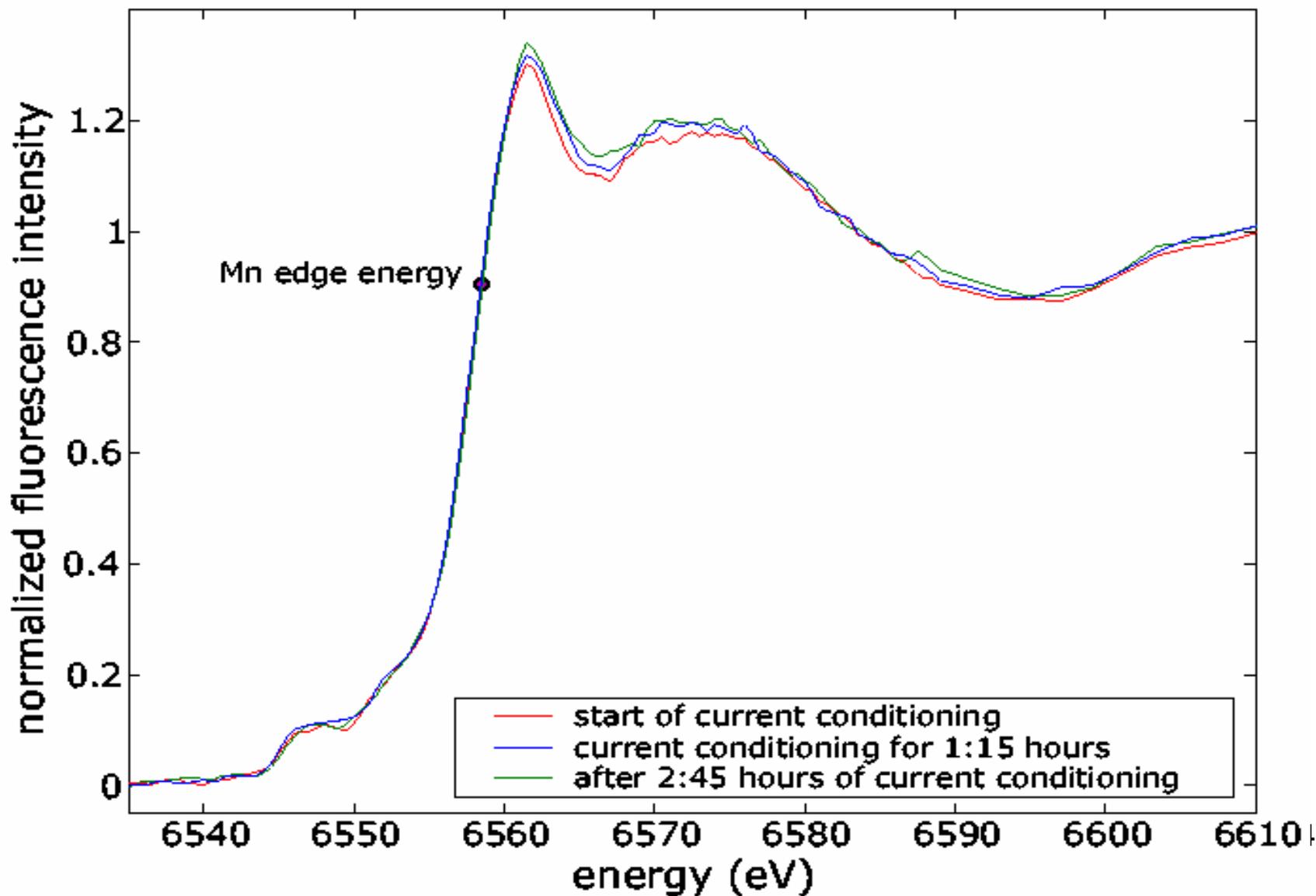


Electrochemical Impedance
Spectroscopy for the electrode area
specific resistance (ASR)



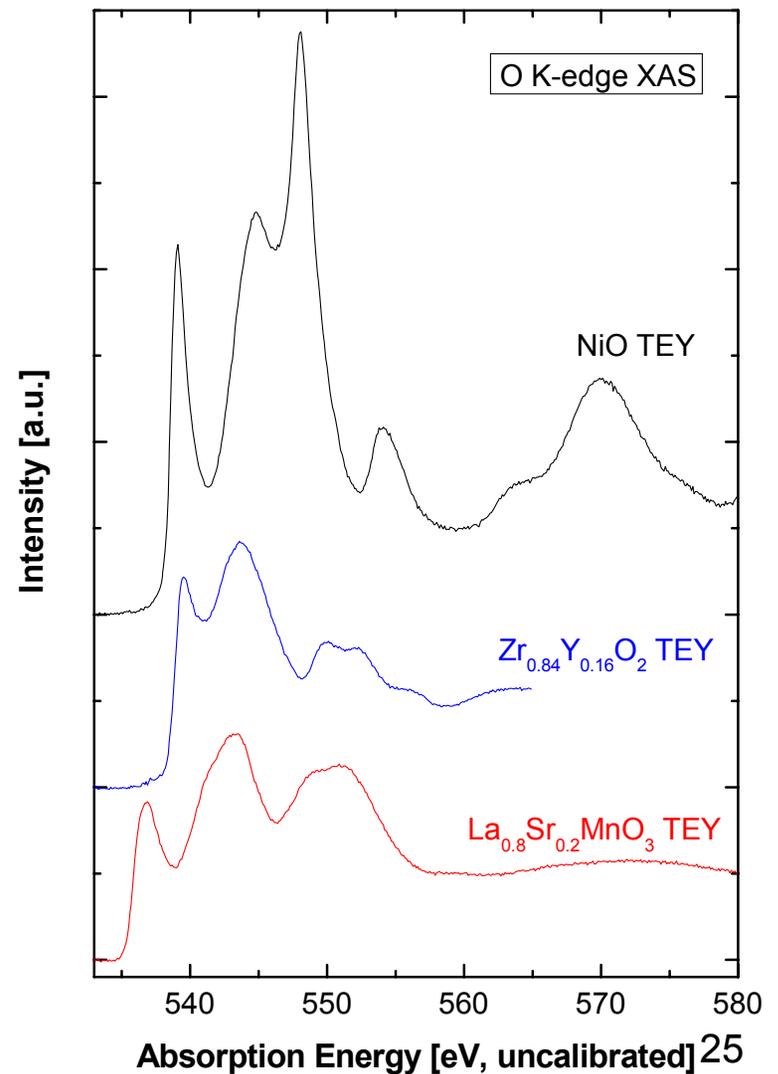
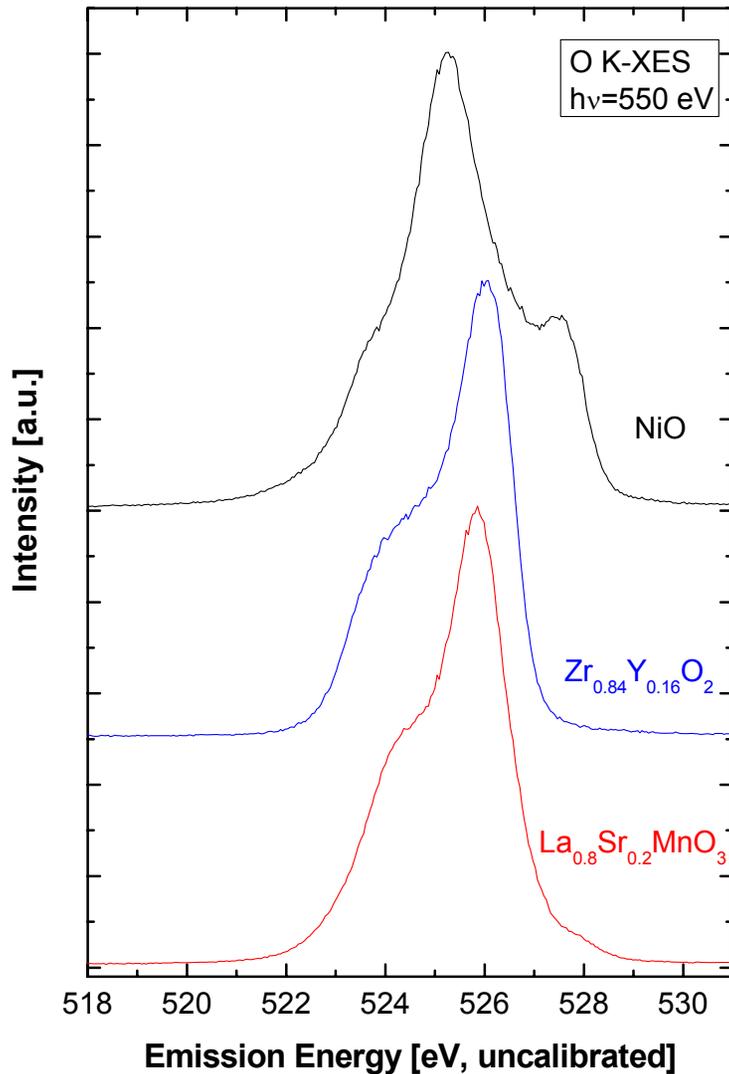
First results with *in situ* cell:

Absorption spectroscopy at the Mn K edge during current conditioning of LSM electrode at -0.6V at 800°C in air



First results:

XES and XAS reference spectra of electrodes and electrolyte



Accomplishments: Task 6: Corrosion and Crack Growth Studies of Materials in HI_x Environment

- Ta-2.5W, Ta-10W and Nb-10Hf were determined to be suitable for high temperature use in HI_x .
- Stress corrosion studies showed the dependence of mechanical properties on environment
- Material test system for three different environments within extractive distillation were completed.
- Ta-2.5W has the best performance in $\text{HI}_x + \text{H}_3\text{PO}_4$.

Long term immersion results

HI_x

| | hours | Corrosion Rate | |
|--------------------|-------|----------------|---------|
| | | mpy | mm/yr |
| Ta-2.5W - 1 | 2072 | 0.0035 | 0.00009 |
| Ta-2.5W - 2 | 1040 | 0.0148 | 0.00038 |
| Ta-10W | 1078 | 0.000 | 0.000 |
| Nb-10Hf | 1120 | 0.036 | 0.0009 |
| Nb-7.5Ta | 1128 | 0.266 | 0.0068 |

$\text{HI}_x + \text{H}_3\text{PO}_4$

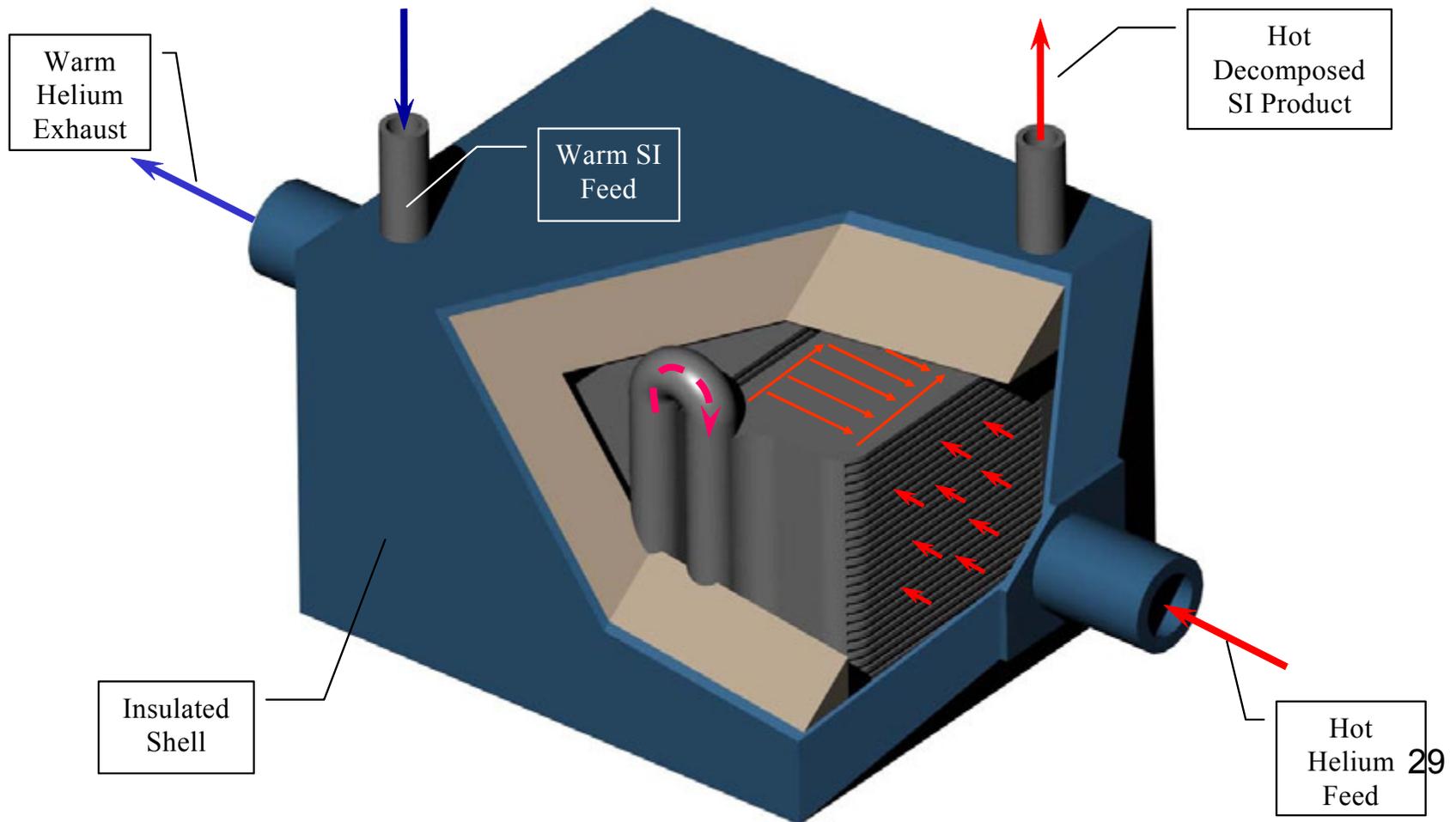
| Sample | Corr. rate (mpy) |
|------------|------------------|
| Nb-1Zr (1) | -0.92 |
| Ta-2.5W | 0.06 |
| SiC | 0.239 |
| Mo | 0.45 |
| Nb-7.5Ta | 22.97 |
| Nb-1Zr (2) | 27.7 |
| Nb | 38.91 |
| Nb-10Hf | 40.49 |
| Zr705 | 91.32 |
| C-276 | 139.88 |
| C-22 | 147.07 |

Accomplishments: Task 7: Ceramic-Based High Temperature Heat Exchanger Development

- Preferred candidate materials tested 1000 hours in steam/H₂SO₄/air environments at 900°C.
- Mechanical Strength: Silicon Carbide & Silicon Nitride – all increased strength; Alumina – decreased strength.
- Weight Gain: Very slight weight gains < 0.1%/1000 hrs. Silicon Carbide & Silicon Nitride form stable “healing” SiO₂ layer. Alumina has corrosion products that are to be determined.
- Compact shell and plate heat exchanger was designed with high performance micro-channels for low volume and low cost.
- Flow/heat transfer coupons designed and fabrication began. Experiments started.
- Thermo-mechanical evaluations are underway.

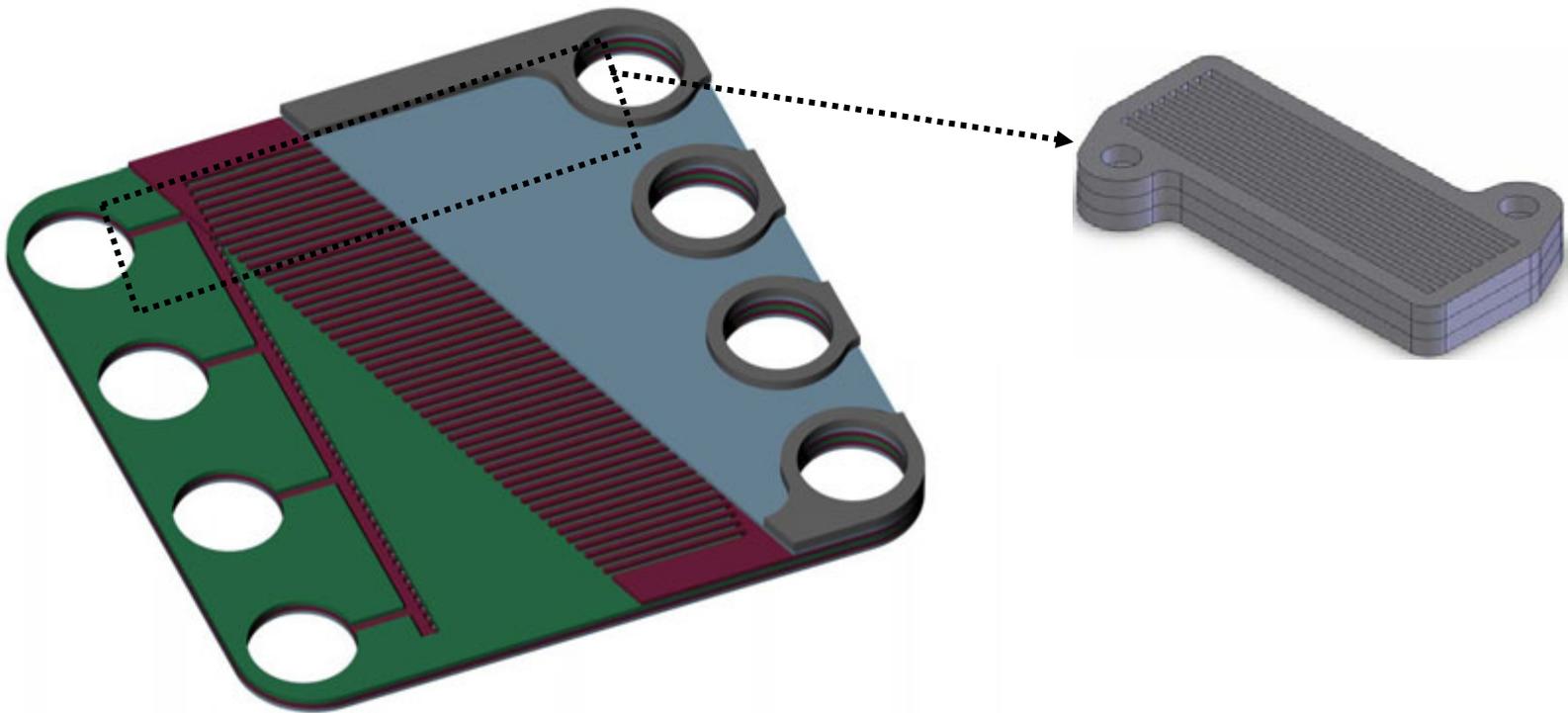
Compact Shell and Plate Design

- High Performance Micro-Channels
- Low Volume, Low Cost



Flow and heat transfer coupons

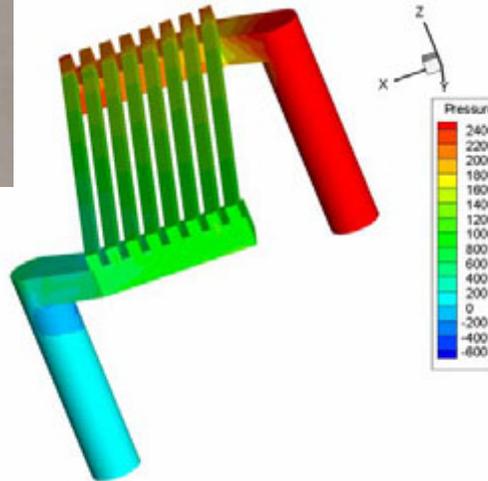
- Sub-section of Micro-Channel Plate Design.
- Flow Dynamics & Heat Transfer Characteristics
- Design & Modeling Validation



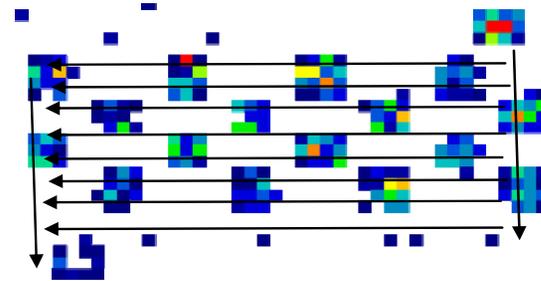
Heat Exchanger Validation

- Coupon Structures
 - Models
 - Conjugate Flow-Heat Transfer
 - Pressure Drop
 - Flow Distribution
 - Experimental
 - Dynamic Sensing
 - Multi-tap Transducer (18)
 - Sensor Averaging

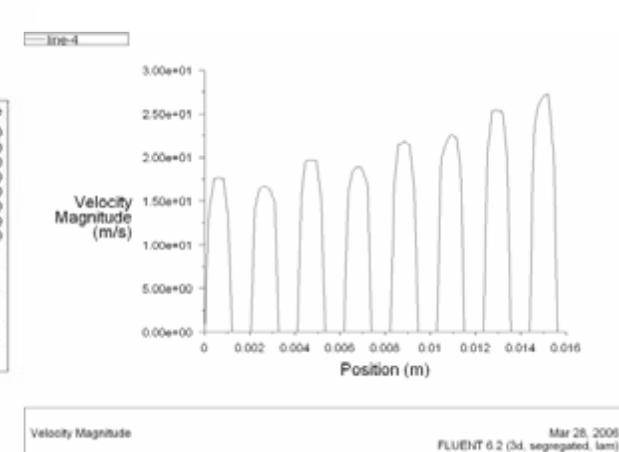
Components



Experimental

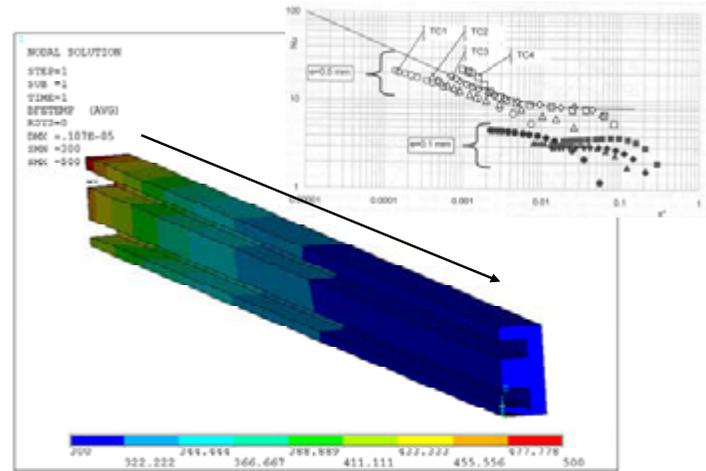
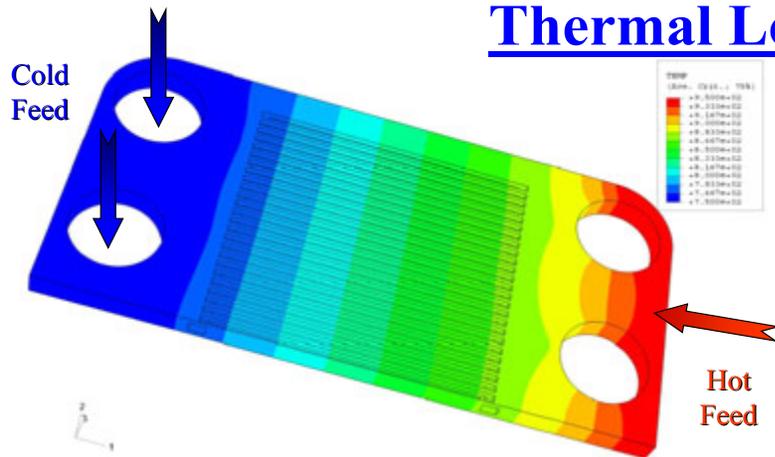


CFD Model

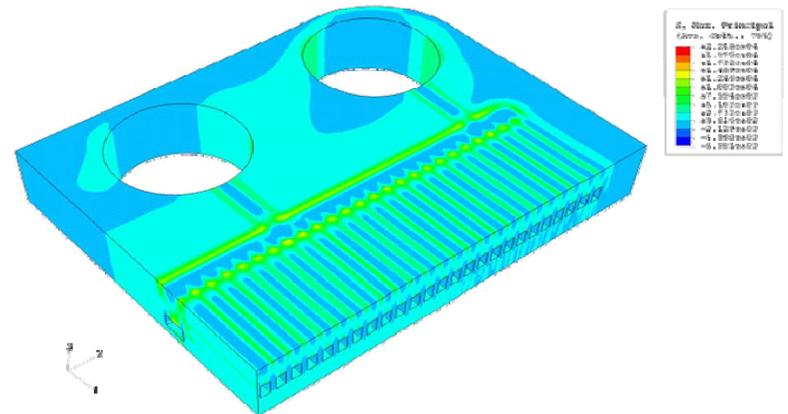
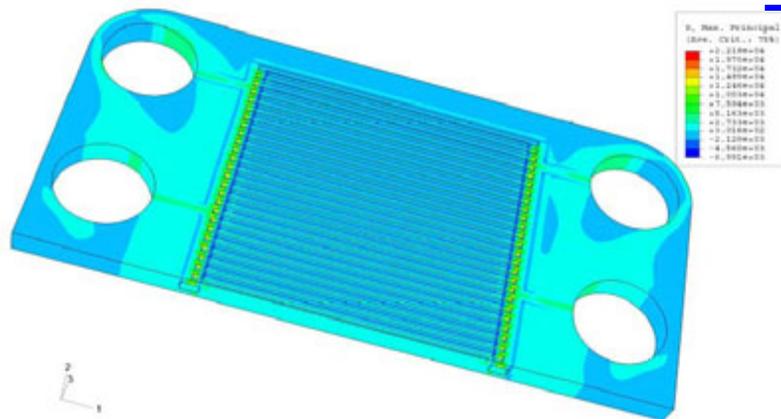


Thermo-Mechanical Design

Thermal Loads



Mechanical Stresses

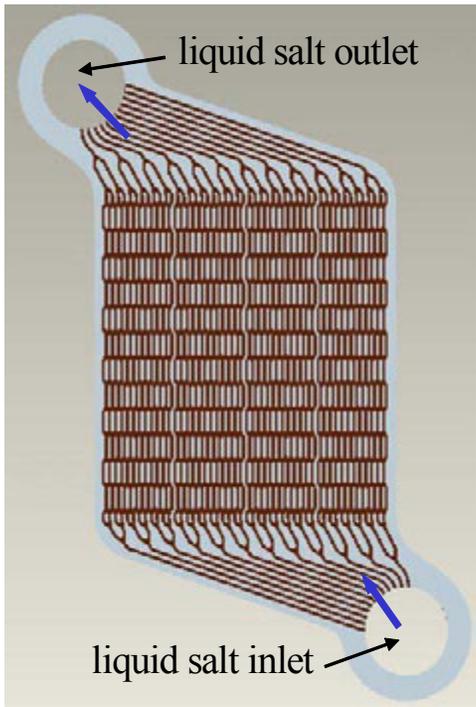


Design Safety Factor = 2.75

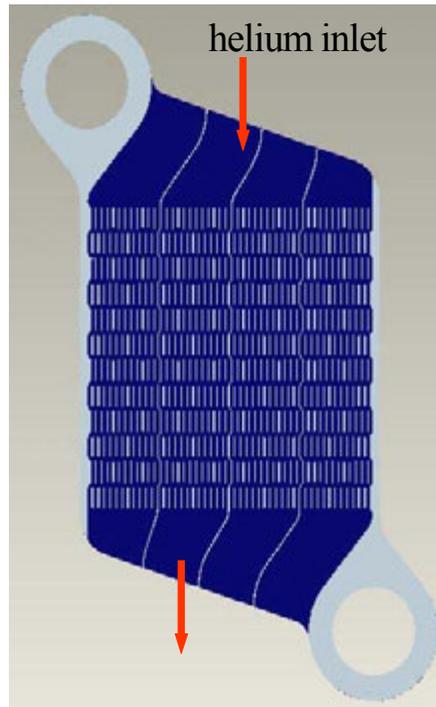
Accomplishments: Task 8: Materials Design and Modeling for C/SiC Compact Ceramic Heat Exchangers

- Completed detailed design for ceramic compact helium-to-liquid-salt HX for thermal/mechanical analysis.
- Demonstrated fabrication of mm-scale fins with thru and blind (preferred) reusable teflon molds.
- Studying use of reactive metal control for flinak coolant to mitigate effects of process fluid ingress.

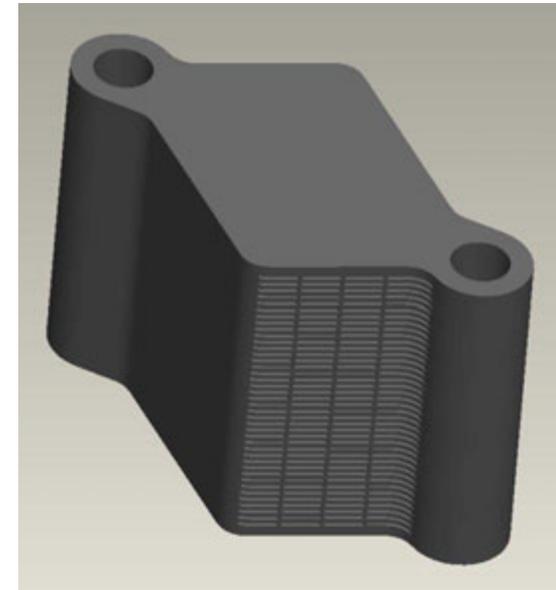
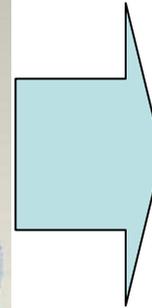
Detailed design for ceramic compact helium-to-liquid-salt HX for thermal/mechanical analysis



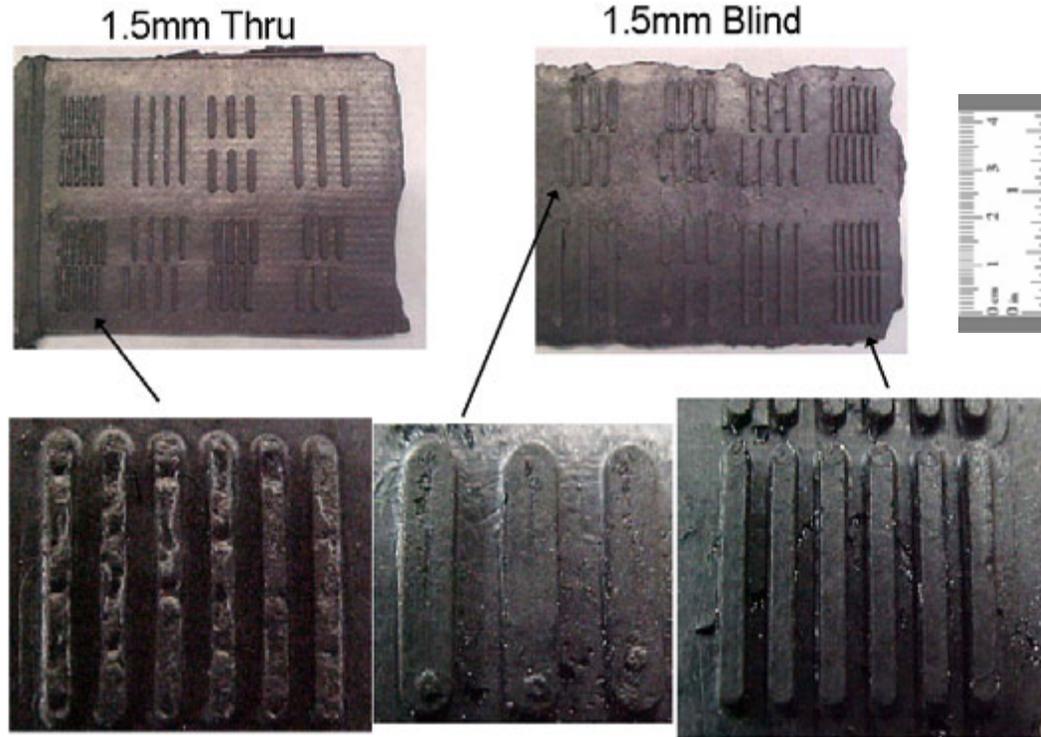
liquid salt side plate



helium side plate



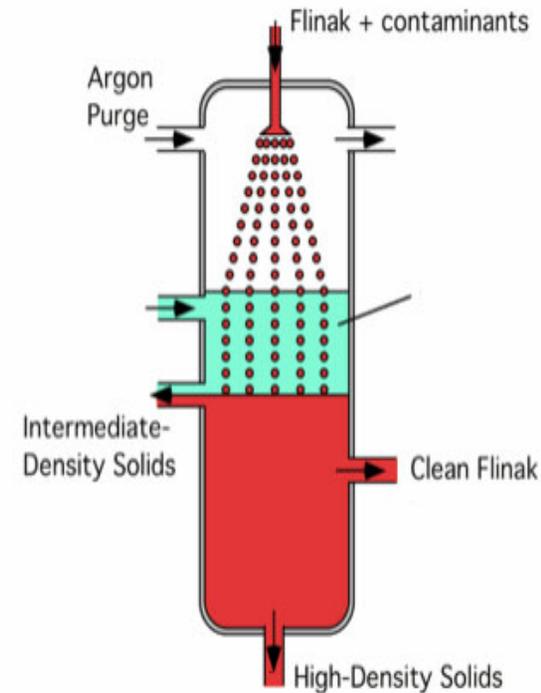
Fabrication of mm-scale fins with thru and blind (preferred) reusable teflon molds



Reactive metal control for flinak coolant to mitigate effects of process fluid ingress



Quartz tube with flinak sample crucible for equilibrium measurement

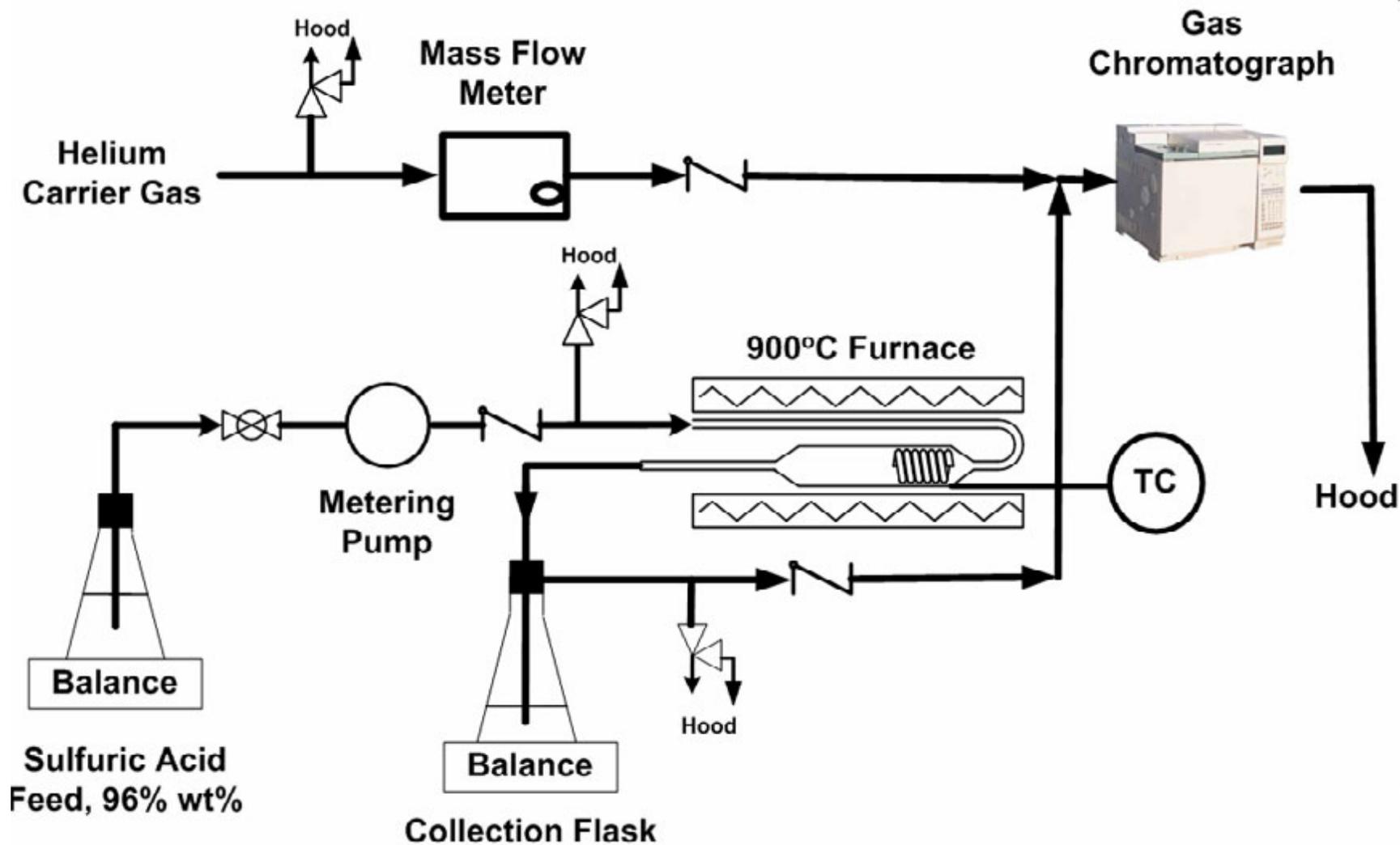


Finite solubility of Na metal in flinak measured, shows potential for this method to be applied for intermediate heat transport for S-I process

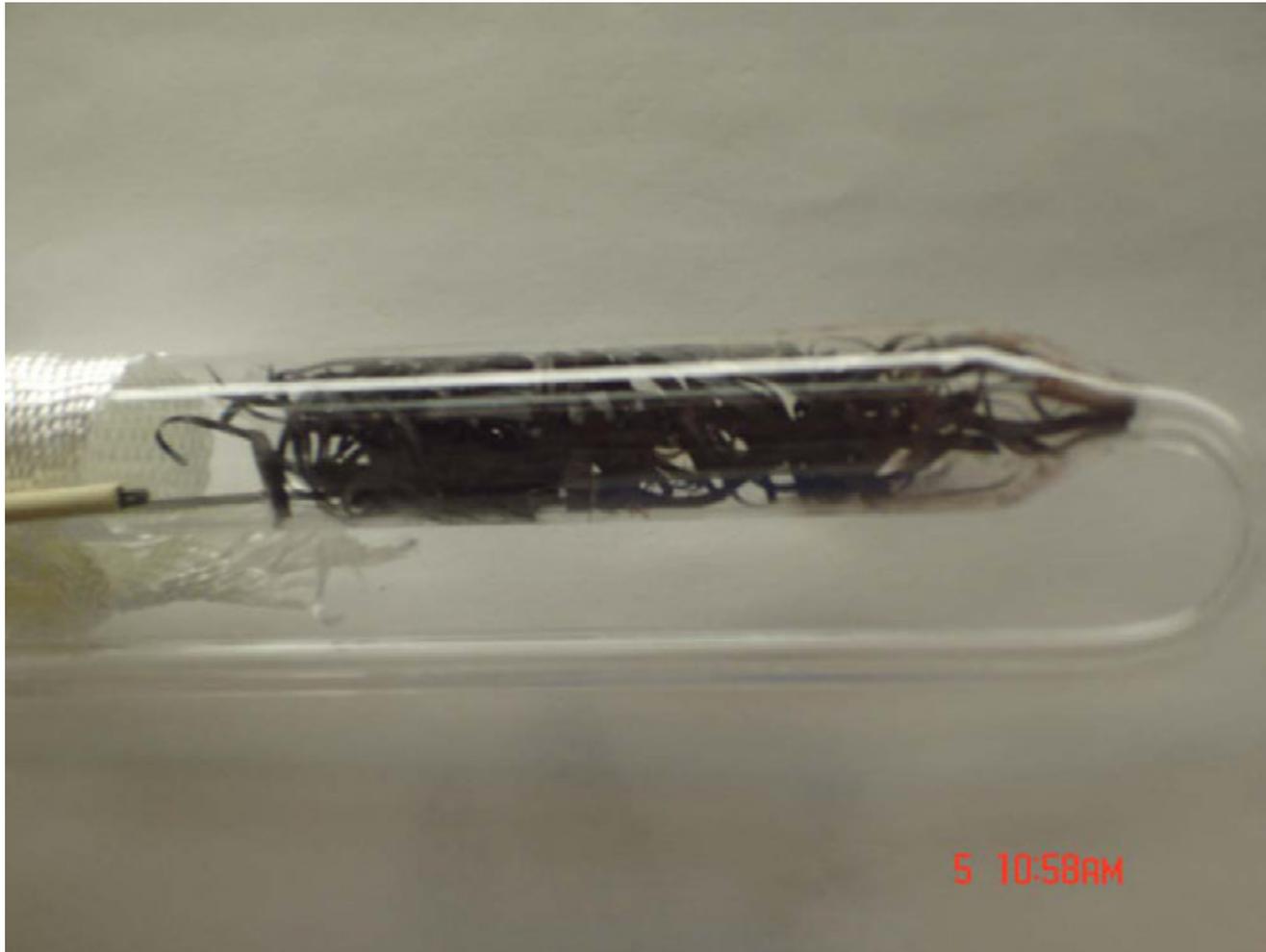
Accomplishments: Task 9: Development of Self Catalytic Materials for Thermo-chemical Water Splitting Using the Sulfur-Iodine Process

- Initial Alloys Produced and Characterized
 - Alloy 800 + Pt
 - Alloy 617 + Pt
- Catalytic Behavior Confirmed
- Catalyst Effectiveness System Construction Finished
 - System In operation
 - Initial Data Developed
- Final Alloy Composition Identified
 - Alloy 800 + 1% Pt
 - Alloy 617 + 1% Pt
- Compact Heat Exchanger Design Finalized
 - Heatric PCHE Design.

Catalytic Test System



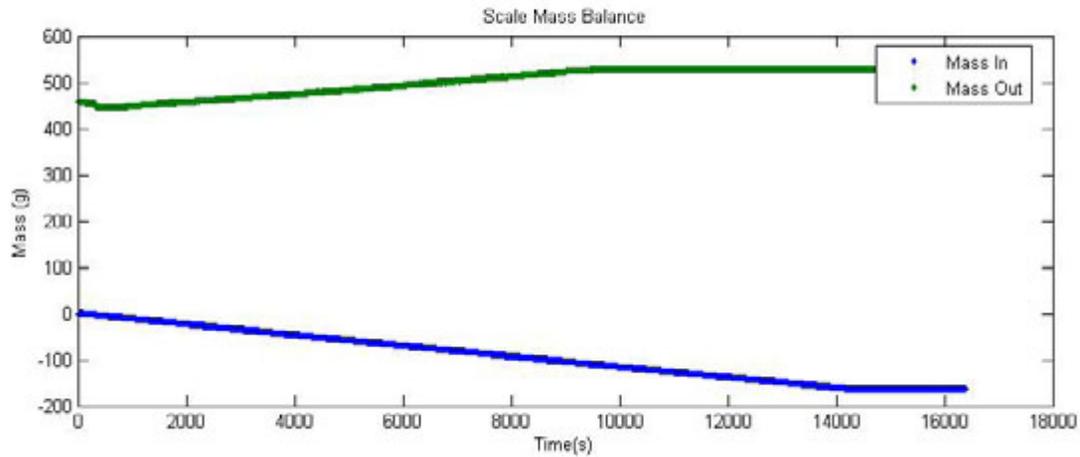
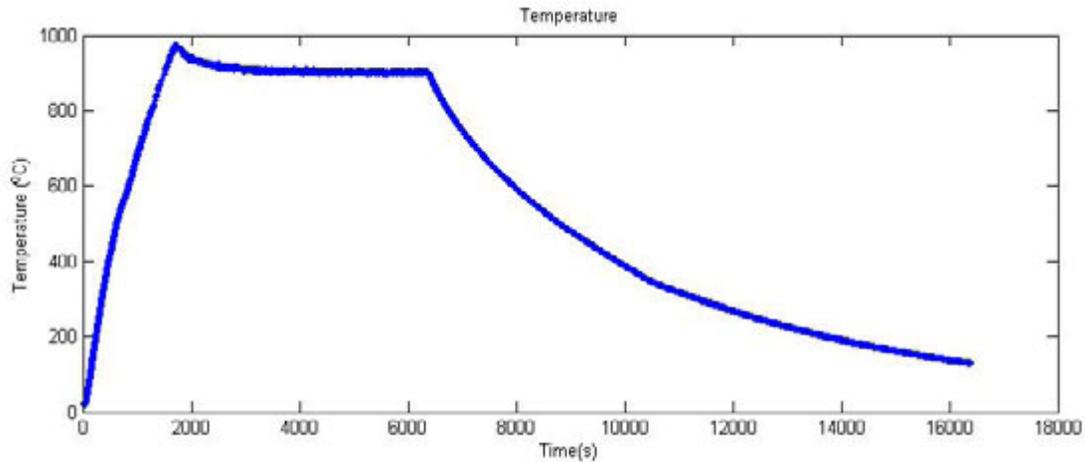
Alloy 800 + 5 wt% Pt-After Test



Student Photo-Working with Catalytic Effectiveness System



Results-Alloy 800 + 5% Pt



Future Work

- Task 1: Heat Exchanger Component Design – Continue numerical analyses for candidate designs, including chemical reactions, and perform optimization studies. Validate numerical results with experimental data.
- Task 2: Identification and testing of candidate metallic materials for heat exchanger components – Evaluate the performance of Alloy 617 and Ta alloys. Perform fracture toughness and crack growth studies of candidate materials. Develop understanding of deformation mechanisms at elevated temperatures.
- Task 3: Heat Exchanger Prototype Testing – Complete thermal and hydraulic testing single-chamber loops. Perform flow visualization. Design and machine double-chamber test sections (counterflow, gas and liquid).
- Task 4: Analytical Studies of the Effects of Acid Exposure on Structure Materials – Continue specimen analyses for materials exposed to HI by General Atomics. Analyze specimens exposed to sulfuric acid by Ceramatec.
- Task 5: Efficiency Improvement and Cost Reduction of Solid Oxide Electrolysis Cells – Next experimental campaign at the Advanced Light Source in May 2006. Conduct surface sensitive x-ray analysis at UNLV. Compare reference LCM electrochemical performance with non-tailored and epitaxially oriented $\text{La}_{0.8}\text{Sr}_{0.2}\text{MnO}_3$ model electrodes.

Future Work

- Task 6: Corrosion and Crack Growth Studies of Materials in Hlx Environment – Complete materials screening for iodine separation, phosphoric acid concentration and HI gaseous decomposition and initiate long term testing. Conduct tests to study the stress corrosion behavior of materials in these environments.
- Task 7: Ceramic-Based High Temperature Heat Exchanger Development – Complete screening corrosion tests for candidate materials. Initiate and execute corrosion parametric tests for baseline materials [f(T,P,C)]. Fabricate and test flow coupons validating the performance expectations. Optimize full-size HX designs based on FEA and experimental results. Fabricate & test performance of full-size components. Develop models for corrosion and mechanical reliability.
- Task 8: Materials Design and Modeling for C/SiC Compact Ceramic Heat Exchangers – Fabricate initial test HX to demonstrate plate lamination. Fabricate test ceramic HX for leak, mechanical and thermal testing to demonstrate utility for IHX application (gas-to-gas, gas-to-LS). Refine volume averaged model method for higher resolution mechanical stress analysis. Develop porous-media-based, volume-averaged modeling method for HX effectiveness and transient thermal stress analysis. Study Na chemistry control methods for flinak experimentally. Study aluminum chemistry for oxygen control in flinak.
- Task 9: Development of Self Catalytic Materials for Thermo-chemical Water Splitting Using the Sulfur-Iodine Process – Make larger heats using commercial practice. Determine mechanical properties of specimens made from larger heats. Complete catalytic effectiveness characterization. Perform further corrosion properties. Fabricate prototype heat exchanger.

Responses to Previous Year Reviewers' Comments

- Questions were fielded and answered by DOE-NE program manager David Henderson.