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High Temperature Heat Exchanger Project

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Quarterly Progress Report
July 1, 2005 through September 30, 2005

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October 31, 2005
1.0 HTHX Project Highlights

- **Quarterly Collaboration Meeting.** The University of California, Berkeley, hosted a UNLVRF HTHX Project quarterly meeting on September 12. The purpose of the meeting was to promote collaboration and communication among the UNLV Research Foundation partners. Collaborators discussed their research program plan and provided an update on their progress. The next meeting will be in December in Las Vegas, NV hosted by UNLV.

2.0 UNLV Design and Testing Group

The University of Nevada, Las Vegas Design and Testing Group supports the following two activities in the UNLVRF High Temperature Heat Exchanger (HTHX) Project:

- HTHX Thermal Systems Design
- Scaled HTHX Tests

2.1 HTHX Thermal Systems Design (PI: Yitung Chen, UNLV)

2.1.1 HTHX Thermal Systems Design Objective and Scope

The HTHX design studies have the following objectives and scope:

- Work with the U.S. Department of Energy Office of Nuclear Energy, Science and Technology (DOE NE) nuclear hydrogen research and development program elements on high temperature systems studies for hydrogen production.
- Identify the range of HTHX applications for Gen IV hydrogen production.
- Develop thermal systems concepts/designs and overall heat/mass balances for the range of Gen IV power conversion and hydrogen production concepts.
- Develop design specifications for the intermediate heat exchanger and other HTHXs used in the conceptual designs.
- Undertake thermal hydraulic systems numerical modeling to establish and analyze temperature, pressure, and flow rate requirements.
- Perform thermal, thermal hydraulic, and structural analyses for selected advanced HTHX concepts for hydrogen production.
- Deliver detailed design for candidate intermediate heat exchanger concepts and materials for hydrogen production requirements.

2.1.2 HTHX Thermal Systems Design Highlights

- **3-D Model Development of an Offset Strip-fin Plate-type Compact HTHX.** Work on optimizing the HTHX design through parametric studies was performed and completed for the gap length and fin length parameters. These were the last 2 of 5 parameters investigated for the off-set strip fin plate-type heat exchanger optimization study. As the gap length increased, the pressure drop decreased, as expected, and no effect on thermal performance was observed. As the fin length increased, the pressure drop decreased and the thermal performance was unaffected.
• **Numerical Analysis based on the Ceramatec High Temperature Heat Exchanger Design for S-I Process – Preheater and Decomposer.** A single channel model of the sulfuric acid decomposition process was used to perform parametric studies on dimensions and flow rates. At this time, only SO\(_3\) decomposition is simulated. Results indicate that the flow rate must be greatly reduced to increase decomposition.

• A single plate model of the heat exchanger was used for studies of flow distribution and pressure drop. The He channel velocity is nearly uniform. The sulfuric acid decomposition channel distribution was found to be very sensitive to flow rate. For a given flow rate, the geometry can be designed using CFD simulations such that the velocities are reasonably similar, e.g., such that the velocities in all the channels do not differ by more than 10%.

• **Sulfuric Acid Decomposition Heat Exchanger Design using Self-catalytic Material.** Simulation cases of SO\(_3\) decomposition using Pt as a catalyst under the experimental conditions provided by Idaho National Laboratory resulted in good agreement with experimental results. A 3-dimensional model was developed to simulate a HEATRIC-type heat exchanger using the self-catalytic material that MIT is developing.

### 2.1.3 HTHX Thermal Systems Design Technical Summary

**Design Optimization Studies for Baseline Offset Strip Fin Compact HTHX:**

Work on optimizing the HTHX design through parametric studies was performed and completed for the gap length and fin length parameters.

*Gap Length:*

The pressure drop values in the liquid salt and helium channels decreased as the gap length was increased, and drastically increased when there was no gap, as shown in Figure 1. This behavior was expected since the inclusion of a gap decreases the magnitude of the flow restrictions present in the flow channel. Also, the presence of a gap in the flow direction does not harm the thermal performance of the heat exchanger, as shown in Figure 2, because vortices still exist in the flow. Therefore, the newly investigated gap parameter can be extremely beneficial in the design of an offset strip-fin heat exchangers.

*Fin Length:*

As the fin length is increased the pressure drop values for the helium and liquid salt channels decrease, as shown in Figure 3. This is attributed to the fact that longer fins mean that there are less of them; thus, effectively decreasing the amount of resistance by the flow through the offset strip-fin heat exchanger. The thermal power on both the helium and liquid salt sides is unaffected by the changes in fin length. The main reason this occurs is that the overall length of the heat exchanger divided by the length of an individual fin is much larger than five. Also, it can be said that the fin lengths studied do not vary significantly. Since the closer one gets to a pin fin arrangement, really short fins, the larger the thermal power values will become, and in the present study the shortest fin is 5 mm long. For the present study a fin of 1 to 2 mm long would represent the pin fin arrangement.
Effect of Gap Length on Pressure Drop

Figure 1. Gap length (mm) vs. pressure drop (kPa) for both He and LS channels.

Unsteady Flow:

An investigation into the need to model unsteady flow in the current HTHX design was studied. A thorough literature review was made, and it was determined that unsteady conditions would more than likely exist in the current HTHX design. The mechanisms responsible for unsteady flow would probably create higher heat transfer rates and pressure drops.

Two dimensional models for both the helium and liquid salt channels were developed. The dimensions were the same as those for the 3-D base cases, and each one was 18 modules long. Simulations using both steady and unsteady flow models were run for the helium and liquid salt channels. The results from the steady flow cases were reasonable, compared to the results from the 3-D base case studies. However, the results from the unsteady flow simulations were not producing the expected results.
For both the helium and liquid salt sides there was no difference between the steady and unsteady models. No change between the steady and unsteady model for the liquid salt side was expected, but a change from the steady to unsteady model for the helium side was anticipated. For the helium side, according to the literature, unsteady flow should be present because the operating Reynolds number is approximately 2,500. Therefore, the unsteady model used was inaccurate, or there is some phenomena present in the HTHX that pushes the critical Reynolds number for the onset of unsteady flow higher. Some of these phenomena may be attributed to the relatively small geometry of the HTHX or the fairly high operating pressure of the HTHX. To determine the reasons why the unsteady model is not working a benchmark test needs to be made. A good case for this would be the unsteady flow over a flat plate or a backward facing step.

**Numerical Analysis Based on the Ceramatec’s High Temperature Heat Exchanger Design for S-I Process – Preheater and Decomposer:**

Parametric studies of the single channel model of the sulfuric acid preheater and decomposer shown in Figure 4 were performed using Fluent version (6.2.16) with modifications made to the solver for the energy equations. The energy balance for the single channel model was improved significantly by using the new version of Fluent.
Two methods were developed that include chemical reaction processes in the single channel model. One includes energy effects of the reaction by increasing specific heat to improve the energy balance of the model. The second method uses a chemical reaction model for the chemical reaction domain.

The results of increasing specific heat, shown in Table 1, improved the energy balance of the model (the temperature gradient in the reaction channel increased, the temperature gradient in the non-reaction part decreased).

The surface chemical reaction model for SO₃ decomposition was applied to the one channel geometry. According to the calculations, the decomposition reaction does not reach the expected value when the inlet condition for velocity of the reactant equals 3.99 m/sec because the flow rate is too large. Based on these results, all of the flow rates will be decreased by ten times to improve conditions for the chemical reaction.
Table 1. Results for different $\Delta C_p$ in the reacting channel.

<table>
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<tr>
<th>Case</th>
<th>Channel</th>
<th>$C_p$, J/(kg-K)</th>
<th>$T_{in}$, K</th>
<th>$T_{out}$, K</th>
<th>$\Delta T (T_{out}-T_{in})$, K</th>
<th>$\Delta P (P_{in}-P_{out})$, Pa</th>
<th>$\Delta Q$, W</th>
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<td><strong>Base case</strong></td>
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<td></td>
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<td>1223.1449</td>
<td>1192.8792</td>
<td>30.2657</td>
<td>488.88998</td>
<td>-0.04491898</td>
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</tbody>
</table>

Note: Specific heat for SiC domain: $C_{p_{SiC}}=1200$ J/(kg-K).
A Pascal code for generating a Gambit journal file was created to conveniently change the channel geometry and mesh in the single plate model of the preheater and decomposer. This significantly shortens the time to get optimal geometry of the plate with a uniform flow distribution for all of the channels in the plate. An example of the geometry is shown in Figure 5. The Y-velocity distribution for the geometry in the middle of the channels is shown in Figure 6. The flow rate differences for all of the channels are not larger than 10%.

It was found that the velocity distribution is very sensitive to the flow rate. Therefore, it is necessary to change the geometry using the Gambit journal file for each flow regime.

Figure 5. Optimized geometry for the plate model.
The sulfuric acid decomposer test coupon geometry was provided by Ceramatec Inc. The coupon will be used for experimental investigations and validation of the Fluent models by comparison with experimental data. The coupon geometry is shown in Figure 7. The Gambit model for all of the six plates of the coupon was created and the Fluent procedure is under development.
Sulfuric Acid Decomposition Heat Exchanger Design:

In past simulation cases, experimental data were not matching simulation results because of variation in different parameters like surface area and mass flow rate. A new set of simulation cases were carried out keeping the conditions close to the experimental work, the results are shown in Figure 8. Both sets of experimental results are from the same source (INL).

![Figure 8. Comparison of percentage decomposition of sulfur trioxide for simulation and experimental results.](image)

The older set of experimental data agrees very well with the simulation results, while the new experimental results start to deviate. A point has been mentioned by the source of the new experimental results that the high temperature yields of sulfur trioxide are lower than expected and the reason could be the deactivation of catalyst as the experiments were run. But in the simulation cases the deactivation is not taken into consideration and so the high temperature yields of sulfur trioxide are as expected and are higher than the new experimental results.

Until now, all the analyses were being carried out on a two-dimensional numerical model. A three dimensional numerical model was used for a MIT designed sulfuric acid decomposer that is a counter flow type heat exchanger with both hot and cold fluids flowing through the tubes. The tubes are piled into a cube of dimensions 0.5m x 0.5m x 0.5m.

The length and diameter of the tubes are 0.5m and 0.0016m, respectively. Helium is considered as the hot fluid which supplies heat to the cold fluid, a mixture of SO$_3$, H$_2$O, SO$_2$, and O$_2$, to perform the chemical decomposition of sulfur trioxide gas. The unit cell of decomposer is shown in Figure 9. A three-dimensional grid has been generated using GAMBIT 2.2 for the unit cell, with around 450,000 nodes. The mesh close to the inner walls of the reactor tubes on the cold
side is finer than at the center because the mesh should be fine enough to capture the wall surface chemical reaction. Since there is no chemical reaction involved in the hot side fluid there is no variation in the mesh from center to the walls. Simulation cases are yet to run on the 3D model.

Figure 9. Unit cell of the sulfur trioxide decomposer taken into consideration for grid generation.

2.2 Scaled HTHX Tests (PI: Samir Moujaes, UNLV)

2.2.1 Scaled HTHX Tests Objective and Scope

The Scaled HTHX Tests have the following objectives and scope:

- It is proposed that an experimental facility be constructed at UNLV. This facility would provide needed experimental validation results to the numerical simulation effort going on within the UNLV Research Foundation consortium. The effort at UNLV will be geared towards the design, testing, and optimization of a High Temperature Heat Exchanger (HTHX) Experimental Facility which will be used as part of the effort by DOE to investigate the possibility of generating hydrogen by thermo-chemical means at high temperatures using heat from a nuclear reactor as the heat source.
- The experimental set up will be a prototype of the actual HTHX. Initially experiment will be run at lower temperatures and with different candidate fluids but with the same flow properties. In final stage the experiment will be run with actual candidate fluids.
- Determining the thermal properties of some of the fluids and solids used in the HTHX.
- Comparing the experimental results with CFD model.

2.2.2 Scaled HTHX Tests Technical Summary

Experimental Heat Exchanger Setup and Testing Design

The main purpose of the experimental apparatus is to test the heat transfer coefficient for the heat exchanger and friction coefficient for the pressure drop of the offset strip fins in the heat
exchanger core. The experiments will be run under the idealizations of steady state conditions, uniform temperature/flow distribution over every flow cross section, and constant specific heat for each fluid.

The testing runs are either a single-chamber setup with one fluid and adjustable electrical heaters or a counterflow double-chamber with different cold and hot fluids flowing through separate chambers. The length scale ratio of prototype to model for the offset strip fin cell is set to be 1:3. The size of the test section is confined by the capacity of pump/fan, heating/cooling elements, available materials, and UNLV machining equipment.

Other than helium, room air will be a testing gas because it will largely simplify the system by eliminating the cooling element as it can be used in a once through system setup. The heat capacity error caused by the humidity will be less than 1%. Distilled-deionized water is considered as another testing liquid because of its well known properties. The Prandtl number of water has the same order of Prandtl number of liquid salts.

The heat transfer coefficient will be evaluated by single-chamber setup and double-chamber setup under the heated conditions. The heating pad/hot fluid should be on the top to avoid natural convection. For the single-chamber liquid setup, the etched foil electrical heater which generates uniform heat flux is applied on the top wall. For the single-chamber gas test section, the heating pad can be applied either on the top wall, or on the bottom wall, or on both walls because the Rayleigh number is small. For the counterflow double-chamber test section, the hot fluid will be heated by the oven first then flows through the top chamber, while the cold fluid will flow through the bottom chamber. The LMTD (logarithmic mean temperature difference) method is chosen for the heat transfer analysis. The bulk mean temperature is used to determine the fluid properties, and the dimensionless parameters.

The friction coefficient here is only evaluated for the core, or the offset strip fin area, and will be tested by the single-chamber setup under heated and isothermal conditions. The friction coefficient will be determined by the effects of skin friction, form drag, and local flow contraction and expansion losses under the isothermal conditions. Because of the wide working temperature range for the prototype heat exchanger, the variable thermal properties of the working fluids are taken into consideration. The heated pressure drop test will provide a guideline for the design.

The control parameters for the experimental work will be flow rate (Reynolds number), fluids’ inlet temperature, and heat flux from the heating pad. When using difference testing fluids, different thermal properties will predominantly affect the testing parameter ranges.

The tests will be run in the low Reynolds number range for the laminar liquid flow. For the gas flow, the tests will be run from high laminar Reynolds number to transitional Reynolds number. The experiment is repeated at different flow rates to cover the desired range of the Reynolds number.
Thermal Property Tests

The solid structure materials of the proposed HTHX properties, density, specific heat, and thermal conductivity, can be tested precisely without difficulties in commercial labs up to 1000°C for the maximum working conditions. Helium properties under working conditions have been measured by different researchers and are well known. Because liquid salts are transparent and corrosive at the working temperature range (550–970°C), the reliability of thermal conductivity tests needs more attention. The density and viscosity can only be tested in labs with very specific techniques. The specific heat can be tested by the modified Differential Scanning Calorimetry. There are some known liquid salt properties in low temperature range in literature.
3.0 UNLV Materials Selection and Characterization Group (PI: Ajit Roy, UNLV)

3.1 Accomplishments

The selection of structural metallic materials and alloys for high-temperature heat exchangers (HTHX) to generate hydrogen using nuclear power source poses a major challenge to scientific and engineering communities. These materials must possess excellent resistance to numerous environment-induced degradation and superior high-temperature metallurgical properties. Three different water splitting cycles namely, sulfur-iodine (S-I), calcium-bromine (Ca-Br) and high-temperature electrolysis (HTE) have recently been proposed to generate hydrogen. A brief description of each cycle has been given in the preceding quarterly reports.

The tensile properties of Alloys C-22, C-276, 800H, and Waspaloy have been evaluated at temperatures ranging between ambient and 600°C using both Instron and MTS equipment. The susceptibility of all four alloys to stress-corrosion-cracking (SCC) has been determined in an aqueous solution containing sulfuric acid and sodium iodide (S-I) using both constant-load and slow-strain-rate (SSR) testing techniques. The localized corrosion behavior of all four alloys has also been evaluated in a similar environment at 30, 60 and 90°C using cyclic potentiodynamic polarization (CPP) method. The effect of applied potential on the SCC susceptibility of all three nickel-base alloys (C-22, C-276 and Waspaloy) has been evaluated in a similar environment using the SSR method. Zr-705, a candidate structural material for use in the Hlx decomposition process, has been tested for evaluation of its tensile properties and SCC resistance using similar testing techniques. Metallographic and fractographic evaluations of the tested specimens have been performed by optical microscopy and scanning electron microscopy, respectively. Two new refractory materials, namely Nb1Zr and Nb7.5Ta have recently been added to the test matrices.

The general corrosion and SCC behavior of all candidate structural materials at elevated temperatures are currently being evaluated inside the autoclave using coupons and self-loaded (C-ring and U-bend) specimens for variable periods. SCC and tensile testing at different temperatures involving Nb1Zr and Nb7.5Ta will be initiated soon. The specimens for fracture toughness and crack-growth evaluations are currently being machined.

3.2 Plans for Next Quarter

- Continue planned experimental work both at UNLV and GA.
- Prepare additional specimens to perform experiments according to the planned matrices.
- Continue literature search.
4.0 University of California, Berkeley (PI: Per Peterson, UCB)

4.1 Objectives and Scope

UCB’s role in the HTHX project is to develop ceramic compact heat exchangers for use in NGNP intermediate loop and hydrogen production loops. The work scope includes: identification and characterization of candidate ceramic heat exchanger materials and processes, identification and demonstration of candidate ceramic heat exchanger fabrication methods, and, design and modeling of high temperature heat exchangers.

4.2 Highlights

- Progress has been made on the vendor support subaward work to COI for making die embossed C/ SiC plates with PIP process.
- Three of four deliverable reports summarizing works on C/SiC heat exchangers design, analysis and material have been finished and formally issued.
- Preliminary whole heat exchanger thermal stress analysis has been finished.
- Experiment to investigate the solubility of NaK in flinak salt was attempted.

4.3 Technical Progress Summary

LSI C-C/Si-C composite and other composite material study

The subaward to COI for vendor support went well. Two sets of graphite molds were fabricated for COI to produce a set of plates to determine the minimum feature size that can be reliably produced. COI uses polymer infiltration and pyrolysis (PIP) process to fabricate chopped carbon fiber reinforced silicon carbide matrix plates. Initial plates have been fabricated. Figure 10 shows the graphite mold and die emboss process. The graphite tool with the various fin features was release coated then filled with the resin-filler mixture. The resin was degassed and then cured under positive pressure. Vacuum was not used during cure to try and minimize pore formation in the resin. In the first try, it was found that it is difficult to release plate from molds. Applying release coating on the mold improved the release but still cannot solve this problem completely. Besides this, porosity in plate material is relative high. Options for reducing porosity have been identified. Figure 11 shows the fabricated plate using new release coating for graphite mold. Except the failure of total release from mold, the plate was successfully fabricated. Very good replication of fin shapes (below 0.5 mm) is shown in the plate. Switching to Teflon molds which is flexible may solve the release problem. Several sets of Teflon molds were fabricated for COI to use.
Thermal design study and review

Preliminary thermal and mechanical analyses were completed for the UCB compact offset fin plate heat exchanger design. The weak points in inlet/outlet manifolds have been identified through the stress analysis, which will be refined in next fiscal year. Finding detailed stress distribution in a complete heat exchanger with direct FEM (Finite Element Method) requires an order of billion FEM computation units and million hours PC computing time. Therefore, it is not practical to analyze the entire heat exchanger design directly. An alternative method was
proposed to obtain approximate stresses that only require a fast PC to finish calculations within a week. The methods are composed of three steps. First, the heat exchanger is broken down into several regions. Unit cell models are built based on each region that captures all of the most important features of that region. The effective mechanical and thermal properties for each unit cell are then founded through FEM simulations. Second, average stress distribution in an overall model composed of various unit cell regions is computed by using the effective mechanical and thermal properties. Third, these average stress values are then applied to the unit cells to find localized points of high stresses. Pro/Mechanica module (Pro/M) in the Pro/E Wildfire Edition is used for FEM stress analysis. Figure 12 shows region division and unit cells. Figure 13 shows samples of global stress and local stress.

![Image of region division and unit cell models for a HX plate.](image)

*Figure 12. Region division and unit cell models for a HX plate.*
Deliverable Reports

The following three of four deliverable reports were completed:

- report on survey of chopped carbon fiber and matrix materials for C/SiC composite heat exchanger;
- report on C/SiC HX thermal and mechanical analysis; and,
- report on C/SiC heat exchanger heat transfer and safety analysis.

The only deliverable report which has not yet been finished is the report on C/SiC composite fabrication methods and test results due to the delay of subaward to vendor support.

Figure 13. Sample global stress and local stress.
5.0 Corrosion Studies of Candidate Structural Materials in HIx Environment as Functions of Metallurgical Variables (Pl: Bunsen Wong, General Atomics)

5.1 Research Progress and Accomplishments

The level 2 milestone (7/15/05) report “Corrosion Screening of Construction Materials for Hydrogen Iodide Decomposition Heat Exchanger Fabrication” was submitted during this quarter.

Task 1: Construction Materials Screening

Milestone 1: completed

Long term immersion testing of Ta-2.5W, Ta-10W, and Nb-10Hf at the boiler condition was conducted during Q4/FY05. The goal is to test the specimens in HIx up to 1000 hrs or until there is no visible change in coupon passivation. Figures 14 and 15 show the progression of the Ta-2.5W coupons immersed in HIx for 1700 and 1040 hours respectively. The samples show no sign of corrosion and change in passivation is relatively minor. The e-beam welds on both specimens also show no sign of corrosion, and only color change due to passivation growth. Hence, Ta-2.5W is a good heat exchanger material candidate. Long term testing of one of the coupons is ongoing and the other is being characterized at UNLV.

Long term immersion corrosion testing (>1000 hours) of both Nb-10Hf and Ta-10W specimens shows that the alloys are stable in HIx. The specimens did not exhibit any sign of corrosion (Figs. 16 and 17). The characteristic of the passivation layer is being investigated at UNLV. Table 2 shows the corrosion rate of the qualified materials after long term testing. The rate is much lower than the initial targeted values: Tubing, Valves – 0.075 mm/yr (0.0004mm/yr actual) in test, Vessel, Pipe – 0.5 mm/yr (0.0004mm/yr actual) in test. Hence, these three materials are good candidates.
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*Figure 14. Ta-10W immersion corrosion coupon with an e-beam weld.*

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*Figure 15. Ta-10W immersion corrosion coupon with an e-beam weld.*
Table 2. Weight change in Ta-2.5W, Ta-10W, and Nb-10Hf samples tested in HI_x.

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</table>
**Task 2: Processing Effects on Corrosion Properties**

*Milestone 2: completed*

A Zr-705 C-ring specimen was tested in the test system that was designed to accommodate larger specimens. This type of specimen can provide data on the stress corrosion behavior of materials in the HI<sub>x</sub> environment. Previous immersion corrosion testing of Zr705 coupons had shown dissolution of material in HI<sub>x</sub>. However, cracks can be found in the C-ring specimens in addition to dissolution which illustrates the effect of stress corrosion. Ta and Nb alloys C-ring specimens are currently being prepared at UNLV. Testing of these specimens is anticipated to begin next quarter.

**Heat Exchanger Material Testing for Extractive Distillation**

In response to the decision of the national S-I program to use the HI extractive distillation, instead of reactive distillation, for the demonstration loop, test systems were designed suitable for testing materials under extractive distillation conditions. Figure 18 shows a flowsheet for the extractive distillation process. The designed systems can test materials of construction for iodine separation, phosphoric acid concentration and gaseous HI decomposition and they are shown in Figs. 19 to 21. An existing test system has been modified for iodine separation material testing. The phosphoric acid test system is being constructed and will be completed during the quarter followed by the gaseous HI decomposition system the following quarter.

Testing for corrosion behavior of material in the iodine separation environment has begun. Figure 22 shows a Zr705 coupon which has been tested in a phosphoric acid-HI<sub>x</sub> mixture at 120°C. Although there is no dissolution, there is clearly a reaction between the chemicals and Zr705. Thus, Zr705 is probably not a suitable construction material for this process step.
Figure 18. Flowsheet for HI decomposition via extractive distillation.
Figure 19. Material immersion test system for the iodine separation environment.

Figure 20. Material test system for the phosphoric acid concentration environment.
Figure 21. Material test system for the HI gaseous decomposition with flowing gases.

<table>
<thead>
<tr>
<th>0 hr</th>
<th>120 hr</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
</tr>
</tbody>
</table>

**Figure 22.** A Zr705 coupon that has undergone immersion coupon testing in the iodine separation environment for 170 hours.

6.1 Introduction

The Sulfur-Iodine process, as it is currently envisioned, will require that an H₂SO₄ decomposition reaction be accomplished over the temperature range from 450-850°C. After decomposition the reaction

\[ \text{SO}_3 \Rightarrow \text{SO}_2 + \frac{1}{2} \text{O}_2 \]

must be promoted using a suitable catalyst. The goal of this task is to develop a heat exchanger that will also be able to serve as a catalyst for the above reaction.

The general approach for the development process is to focus on an alloy system that would normally be considered for the acid decomposition reaction and to modify this chemistry via the addition of a catalytic element. The program is focusing on alloys-800HT and alloy 617 to which Platinum has been added to the base chemistry. Several alloy chemistries will be produced, first in small “button” quantity form, and then in larger small heat size form that can be fabricated into useful shapes for characterization and analysis-both metallurgically/mechanically and for catalyst effectiveness. Lastly, depending on the results of the initial development process, material will be used to fabricate a small heat exchanger for actual testing.

This task is being performed in 4 subtasks: (1) Material chemistry identification, procurement and metallurgical characterization, (2) Catalyst effectiveness determination, (3) mechanical properties determination and (4) prototypic shape fabrication and testing. In this report tasks are indexed to the larger consortium heat exchanger project in which the catalyst work is identified as Task 3.

6.2 Progress

In this document results of the program are reported for the Quarter July 1-September 30, 2005. Reporting will be by subtask as identified in the master proposal for the program.

Subtask 3.1: Material Chemistry Identification, Alloy Procurement and Metallurgical Characterization

Subtask 3.1.1: Initial Chemistry Identification & Characterization

This subtask was completed during the previous quarter. A series of alloy 800HT plus Pt and alloy 617 plus Pt alloys in “button” form have been melted and characterized from a metallurgical standpoint. As a result of this characterization, the chemistry of the larger heats have been defined. The larger heat chemistries will consist of 2 wt% or less Pt added to base chemistries for alloys 800 and 617.
**Alloy Development**

**Subtask 3.1.2: Larger Size Quantity Production**

The chemistries of the larger heats have been defined and cost estimates for their production have been developed. These heats will be produced in FY06 using funds from the FY06 budget for the project.

**Subtask 3.1.3: Powder Production**

No progress was made on this task during this reporting period.

**Subtask 3.2: Catalyst Effectiveness Determination**

**Subtask 3.2.1: Facility Construction**

The catalyst effectiveness system has been designed and procurement is complete for the major components. Figure 23 shows a schematic of the system. H$_2$SO$_4$ will be boiled and decomposed in a high temperature furnace @ 900°C during an initial pass through the furnace. The second pass will take the vapor (acid/He mix) over the catalyst where the SO$_3$ to SO$_2$ reaction will be catalyzed. The exit gas stream, containing water vapor, SO$_2$, O$_2$ and any remaining SO$_3$ will then be stripped of the SO$_3$ remaining and the oxygen removed. The remaining stream will then pass through the Gas Chromatograph where the SO$_2$ concentration will be measured. Any excess SO$_2$ and O$_2$ will then be recombined and processed to H$_2$SO$_4$.

**Subtask 3.2.2: Catalyst Proof of Principal**

During this reporting period material from the “button” heats were sent out to be processed into thin foil material for testing in the catalyst effectiveness system. This material is in process. Once the material has been received it will be used to “shake down” the catalyst effectiveness system and to evaluate the initial effectiveness.

**Subtask 3.2.3: Catalyst Effectiveness**

**Subtask 3.3 Mechanical Properties Determination**

No progress was made on this task during this reporting period.

**Subtask 3.4 Prototypic Shape Fabrication and Testing**

**Subtask 3.4.1: Compact Heat Exchanger Application**

This subtask is in the initial design phase for the test article heat exchangers with the Heatric company regarding the manufacture of small heat exchanger modules using one or more of the alloys being developed in the program.
**Figure 23. Schematic of Catalyst Effectiveness System.**

The Heatric Company was provided with the requirements for the test modules and the design is being finalized. The exact product form required for the heat exchanger fabrication has been defined. The Special Metals Company, the supplier of the larger heats of material, has been included in the design process and will provide the proper product form to Heatric for the heat exchanger fabrication. Heat exchanger fabrication will take place as a part of the FY07 work. However, since the larger test heats of catalytic material will be produced in FY06 (and processed into plate form for heat exchanger fabrication) a base design was needed for the heat exchanger itself although actual fabrication will not take place until FY07. Two basic types of heat exchanger have been considered: (1) the so-called printed circuit heat exchanger and (2) the diffusion bonded formed plate-fin heat exchanger. The latter design was settled on. Figure 24 shows a representation of this design. This design was chosen because with this design it is possible to use laminated sheet (one side containing the Pt alloyed material and the other being the corresponding non-Pt containing alloy) and reduce costs.
Figure 24. Diffusion bonded plate fin heat exchanger design.

Subtask 3.4.2: Shell & Tube Heat Exchanger

No progress was made on this task during this reporting period.
7.0 Development of an Efficient Ceramic High Temperature Heat Exchanger (PI: Merrill Wilson, Ceramatec, Inc.)

7.1 Program Scope and Objectives

The objective of this research is to assess the technical feasibility and economic viability of using ceramic and/or ceramic composite based materials for high temperature heat exchangers. The technical feasibility will be addressed through:

- Materials design, wherein the corrosion, mechanical and thermal properties of preferred materials will be evaluated, and
- Heat exchanger design, wherein the heat, mass and mechanical design issues are numerically modeled and validated through empirical testing.

The economic viability will be assessed through cost models based on the above designs and common ceramic manufacturing practices.

7.2 Program Highlights

- Two high temperature exposure test rigs have been designed, equipment for them ordered, and assembly begun. The first test rig is designed to expose ceramic samples to steam/oxygen atmospheres such that their baseline corrosion rates can be measured and compared to literature data. The second test rig will be capable of introducing sulfuric acid and thus quantify the enhanced corrosion rates due to the acid. Each test rig is capable of testing about fifty 4 point bend bars samples simultaneously to temperatures of about 1000°C at one atmosphere pressure.
- An updated cost model was completed for the gas to gas heat exchangers in the sulfuric acid decomposer. These preliminary estimates provide a benchmark whereby design improvements/refinements can be gauged.
- A thermo-mechanical analysis has been completed in order to estimate the design reliability and durability of the micro-channel based structure. These results indicate that the design conditions ($\Delta T<25^\circ$C, channel width = 1270 micron) are well within the stress limits and failure probability for either 15 atmosphere or 70 atmosphere process cycles.
- A thermal/flow test coupon has been established and fabrication of these components has begun.

7.3 Research Accomplishments

Task 1 Materials Design Workplan

Ceramatec procured strength/corrosion sample bars for most of the candidate materials. Specimens for measurement of thermal diffusivity are being prepared and will be sent to Idaho National Laboratory (Dr. T. Lillo) in early September. Table 3 summarises the sample inventory. Test plans for materials and conditions for screening potential materials and characterizing candidate materials were developed and are shown in Tables 4 and 5.
Table 3. Inventory of strength/corrosion specimens.

<table>
<thead>
<tr>
<th>Material</th>
<th># of Specimens</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>80</td>
<td>Ceramatec sintered SiC</td>
</tr>
<tr>
<td>SiC</td>
<td>124</td>
<td>Morgan, Inc., sintered SiC</td>
</tr>
<tr>
<td>Si$_3$N$_4$ (hot pressed)</td>
<td>100</td>
<td>Ceradyne material</td>
</tr>
<tr>
<td>Si$_3$N$_4$ (gas pressure sintered)</td>
<td>130</td>
<td>Ceradyne material</td>
</tr>
<tr>
<td>SiAlON</td>
<td>8</td>
<td>Vesuvius material – expensive raw material</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>50</td>
<td>Ceramatec powder pressed</td>
</tr>
<tr>
<td>Other materials (MoSi$_2$, Ti$_3$SiC$_2$, Cordierite)</td>
<td>None as yet</td>
<td>Lower priority – lower material, mechanical, corrosion properties. Vendors being solicited.</td>
</tr>
</tbody>
</table>

Table 4. Preliminary Screening of ceramics performed at Ceramatec, Inc.

<table>
<thead>
<tr>
<th>NHI Application</th>
<th>Temperature</th>
<th>Environment</th>
<th>Materials</th>
<th>Evaluation Metrics</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI Decomposition</td>
<td>950°C</td>
<td>60% Steam</td>
<td>Silicon Carbide</td>
<td>- Weight gain/loss</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30% H$_2$SO$_4$</td>
<td>Sintered SiC</td>
<td>- Strength</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10% O$_2$</td>
<td>RB SiC</td>
<td>- SEM (microstructure)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>CVD SiC</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Silicon Nitride</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• Sintered Si$_3$N$_4$</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• Hot-pressed Si$_3$N$_4$</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• HiPed Si$_3$N$_4$</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• CVD Si$_3$N$_4$</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>SiAlON</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>MoSi$_2$</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Alumina</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cordierite</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Mullite</td>
<td></td>
</tr>
<tr>
<td>Oxygen Recovery (high temperature electrolysis)</td>
<td>850°C</td>
<td>60% O$_2$</td>
<td>Alumina</td>
<td>- Weight gain/loss</td>
</tr>
<tr>
<td></td>
<td></td>
<td>40% Steam</td>
<td>Zirconia</td>
<td>- Strength</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hafnia</td>
<td>- SEM (microstructure)</td>
</tr>
<tr>
<td>Oxygen Recovery (SI process)</td>
<td>900°C</td>
<td>60% Steam</td>
<td>Electrode and Electrolyte Materials</td>
<td>- Weight gain/loss</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30% H$_2$SO$_4$</td>
<td></td>
<td>- Strength</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10% O$_2$</td>
<td></td>
<td>- SEM (microstructure)</td>
</tr>
</tbody>
</table>
Table 5. In-Depth Characterization of ceramics performed at Ceramatec, Inc.

<table>
<thead>
<tr>
<th>NHI Application</th>
<th>Temperature</th>
<th>Environment</th>
<th>Evaluation Metrics</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI Decomposition</td>
<td>a) 950°C</td>
<td>1) 60% Steam, 30% H₂SO₄, 10% O₂</td>
<td>- Weight gain/loss</td>
</tr>
<tr>
<td></td>
<td>b) 850°C</td>
<td>2) 60% Steam, 30%H₂SO₄, 10% Ar</td>
<td>- Strength</td>
</tr>
<tr>
<td></td>
<td>c) 750°C</td>
<td>3) 60% Steam, 30% Ar, 10% O₂</td>
<td>- SEM (microstructure)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>- Hermiticity (He leak rates)</td>
</tr>
<tr>
<td>Oxygen Recovery</td>
<td>a) 950°C</td>
<td>1) 60% O₂, 40% Steam</td>
<td>- Weight gain/loss</td>
</tr>
<tr>
<td></td>
<td>b) 850°C</td>
<td>2) 60% O₂, 40%Ar</td>
<td>- Strength</td>
</tr>
<tr>
<td></td>
<td>c) 750°C</td>
<td>3) 20% O₂, 80% Ar</td>
<td>- SEM (microstructure)</td>
</tr>
<tr>
<td>Oxygen Recovery (SI process)</td>
<td>a) 950°C</td>
<td>1) 60% Steam, 30% H₂SO₄, 10% O₂</td>
<td>- Weight gain/loss</td>
</tr>
<tr>
<td></td>
<td>b) 850°C</td>
<td>2) 60% Steam, 30%H₂SO₄, 10% Ar</td>
<td>- Strength</td>
</tr>
<tr>
<td></td>
<td>c) 750°C</td>
<td>3) 60% Steam, 30% Ar, 10% O₂</td>
<td>- SEM (microstructure)</td>
</tr>
</tbody>
</table>

The flexural strength of two candidate materials was tested, for comparison with retained strength after corrosion testing. The strength of sintered silicon carbide manufactured by another commercial vendor, Morgan, Inc., had an average strength of 359 MPa with a 95% confidence interval of 28.6 MPa. These data will be used to compare with silicon carbide produced at Ceramatec that will be made by the tape cast process that will be used to fabricate heat exchanger plates. The average flexural strength of samples of hot-pressed silicon nitride, manufactured by Ceradyne, Inc., was 710 MPa. Testing of other candidate materials will be performed in early October.

A “dry run” for the sulfuric acid exposure test rig was completed. The data indicates that we should have a fairly uniform heated zone and will be able to test at approximately 950°C. Facility modifications are underway as part of the safety commissioning of the sulfuric acid exposure test rig. These modifications include a primary cooling loop to condense gaseous sulfuric acid from exhaust stream and secondary, an enclosure with exhaust system to prevent uncondensed species from entering the room in case of failure. The goal is to do exposure tests by the end of September.

**Task 2 High Temperature Heat Exchanger Design and Validation**

*Heat Exchanger Cost Modeling* – The cost model was updated for the gas to gas heat exchangers used for sulfuric acid decomposition as per the General Atomics flow sheet. These models are based on the currently practiced Laminated Object Manufacturing (LOM) method at Ceramatec, Inc. These manufacturing and scale-up estimates were derived from outside consultants (CKGP) who specialize in the automation and scale-up of manufacturing plants. Using their cost models and assuming that the aggregate production rate for similar heat exchangers totaled to be 1,000 units per year, a price for the Sulfuric Acid Decomposer (gas-to-gas heat exchanger portion) was obtained. These estimates are preliminary and can be verified as the design is refined and as
prototypical heat exchangers are manufactured. Included within these cost models are the capitalization and facilitization of a new manufacturing facility capable of producing the aggregate 1,000 heat exchangers per year. The direct costs (labor, materials and consumables) and the indirect costs (management, benefits and overhead) are based on current labor and material costs for bulk manufacturing processes. Additional expenses were included to assemble these modular stacks into a high pressure, internally insulated pressure vessel acting as the shell side of the process. It should also be noted that these estimates are based on a projected price where a 15% fee is assumed as the profit margin above the other expenses. The cost structure for the heat exchanger is described in Table 6.

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Direct Labor</td>
<td>Labor for value added fabrication processes</td>
<td>$11,603,130</td>
</tr>
<tr>
<td>Indirect Labor</td>
<td>Labor for management and QC in fabrication</td>
<td>$13,550,160</td>
</tr>
<tr>
<td>Benefits</td>
<td>For both direct and indirect labor (30% of labor)</td>
<td>$7,545,990</td>
</tr>
<tr>
<td>Overhead</td>
<td>Facilities, maintenance and utilities (20% of labor)</td>
<td>$6,539,860</td>
</tr>
<tr>
<td>Sub-total</td>
<td>Labor related variable costs</td>
<td>$39,239,140</td>
</tr>
<tr>
<td>Direct Materials</td>
<td>Material expenses directly used in product (SiC powder)</td>
<td>$5,878,150</td>
</tr>
<tr>
<td>Consumables</td>
<td>Materials used to produce product (Mylar, solvents etc)</td>
<td>$3,994,700</td>
</tr>
<tr>
<td>Heat exchanger vessel</td>
<td>Finishing, packaging, insulating heat exchanger ($20,000 each)</td>
<td>$20,000,000</td>
</tr>
<tr>
<td>Module/Vessel Assembly</td>
<td>Insertion &amp; Plumbing of HX Modules</td>
<td>$12,500,000</td>
</tr>
<tr>
<td>Sub-total</td>
<td>Materials related fixed costs</td>
<td>$42,372,850</td>
</tr>
<tr>
<td>Capital Depreciation</td>
<td>10 year straight-line (equipment, facilities)</td>
<td>$10,318,775</td>
</tr>
<tr>
<td>Sub-total</td>
<td>Annual expenses</td>
<td>$91,930,765</td>
</tr>
<tr>
<td>Management</td>
<td>Corporate G&amp;A (20% of expenses)</td>
<td>$18,386,150</td>
</tr>
<tr>
<td>Sub-total</td>
<td>Cost of production</td>
<td>$110,316,915</td>
</tr>
<tr>
<td>Profit</td>
<td>15% of production costs</td>
<td>$16,547,540</td>
</tr>
<tr>
<td>Total Revenue</td>
<td></td>
<td>$126,864,455</td>
</tr>
<tr>
<td>Price per Unit</td>
<td>Total/1,000 per year</td>
<td>$126,864</td>
</tr>
</tbody>
</table>

Table 6. Price Estimates for the Gas-to-Gas portion of the Sulfuric Acid Decomposer.

Heat Exchanger Design and Analysis – The design of the heat exchanger plate internal manifolds has undergone further design iterations in order to improve the flow distribution across the channels, see Figure 25. The basic design concept is that of using a two-stage header that can redistribute the flow effectively before entering the micro-channels. The design parametrics included the number, location and dimensions of interconnecting vents. The numerical analyses run by UNLV indicate that the flow can be variability in flow rates can be kept within about \( \pm 15\% \) of the nominal condition.
A thermo-mechanical analysis was done for the heat exchanger design in order to estimate the reliability/durability during steady-state operation. The objective of this task was to analyze the micro-channel network design to ensure a reliable design for the heat exchanger plates. These analyses included the effects of both the possible mechanical loads due to pressure differential and the thermal gradients within the heat exchanger plates. This work was to provide design rules such that design modifications could be made and validated through empirical and analytical methods.

Due to the brittle nature of ceramic materials, the reliability of components is subject to critical flaw size and distribution statistics. These statistics are based on empirical testing of analog fracture specimens (typically, 4 point bend bars) where failure modes and conditions can be well characterized. From these data, reliability extrapolations can be made through stress models (finite element analysis) and integration of these stresses over the component.

For this task the stresses induced in the region containing micro-channels was calculated and integrated to estimate the reliability of the heat exchanger plates. The loading mechanisms considered were the pressure loads due to the maximum possible differential pressure (either 15 or 70 atmospheres) and the thermal gradient (<25C) through the solid due to heat transfer. These assumptions should capture the worst conditions when operating at steady state ad an upset occurs (depressurization of one side). Figure 26 describes the model used in calculating the stresses in the micro-channel region. Also included with this figure is the mathematical formulation for computing the reliability of the stressed body.
These calculations were carried out parametrically in order to determine the sensitivity and the limits of the channel width (500-2000 micron), pressure differential (15 and 70 atmospheres) and thermal gradient 25-100°C. From the calculations, the tensile stresses were integrated over the volume of the micro-channel region of the plate. These integrated stresses were then scaled by the Weibull parameters to estimate the reliability of a system. Although arbitrary, it was assumed that for this application the probability of a system failure must be less than 1/1000. This stringent criterion effectively reduces the allowable design stress by several orders of magnitude which can be correlated to the Weibull Material Scale Parameter. Also in order to achieve a low probability of failure the Weibull Modulus must be large. This parameter is similar to the standard deviation for a normal distribution and reflects the consistency and repeatability of the material strength. This can only be achieved in ceramics through consistent and repeatable manufacturing processing. The results of these calculations are shown in Figure 27.

These results indicate that at higher pressures (solid lines) wide channels are infeasible or nearly infeasible at any thermal gradient condition. This is shown by the relatively flat sloped curves where the bending stresses dominate the failure mechanism. However, when narrow micro-channels are used in the design (<1500 micron), the bending stresses are minimized sufficiently and permit steeper thermal gradients (and hence stresses) to occur in the heat exchanger plates. As seen in this figure, if the micro-channels are narrower than 1500 micron, either pressure differential (15 and 70 atmosphere) and thermal gradients less than 60°C through the membrane yield feasible solutions. This indicates that this micro-channel device will tend to be very robust; a pressure trip should not “shock” the system and initiating and sustaining a 60°C through the thickness of the heat transfer membrane (380 micron) is nearly impossible. (This translates to a flux beyond 6MW/m²). It should also be noted that the most extreme thermal gradient (16°C) predicted from the thermal/flow calculations was found only locally near the flow entrance when the thermal conductivity of SiC was reduced an order of magnitude (40W/mK to 3 W/mK). Thus, the assumed design condition, a thermal gradient of 25C with 1270 micron channel width, corresponds to a probability of failure about 100 to 10,000 times less than the design allowable for 70 atmospheres and 15 atmospheres pressure differential respectively.
This “Margin of Safety” is reasonable this early in the design and validation stage. As the design progresses and analytical methods capture all the coupled loading mechanics, this “Margin of Safety” may tend to decrease.

Heat Exchanger Design and Validation – The objective of this task was to empirically validate the thermal and flow performance of the micro-channel design. This task has two motivations a) assist and improve the micro-channel design and performance, and b) use this empirical data to calibrate the numerical models leading to full-scale performance predictions.

Although the end objective to design and validate the design of a full-size heat exchanger plate, the effort and turn-around for these larger components suggests that a sub-scale coupon would be more time and budget economical. Thus a design for a thermal/flow test coupon that can be easily modified to parametrically analyze design and performance variables has been achieved. This test coupon represents a slice of the full-size design, thus including multiple channels yet eliminating the need for flow distribution headers. In this manner we will be able to measure the pressure in and along the channels and the temperature gradient down the axis of the flow channels. This design has sufficient flexibility to modify the height, width and number of channels. It is also large enough to address flow distribution within the manifolds. The design of the thermal/flow coupon is found in Figure 28.
Currently these thermal/flow coupons are being fabricated to be used for testing in FY06 (see Figure 28). The process for fabricating these coupons follows that as described earlier (LOM process). However, for the coupons we are fabricating them in a “3-up” configuration which will more closely mimic the lamination conditions and tooling required for the full-size heat exchanger plate. Thus, worked out and QC monitored processes and equipment should be able to be used when migrating back to the full-sized design. The layers, tooling and lamination press are shown in Figure 29.
Figure 29.  a) Lamination Press, b) Lamination Die, c) Micro-channel Slots (3-up), d) Manifold Slots (3-up), e) Laminated Wafer (1/2 complete), and f) Laminated Wafer with Enclosed Features.