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Manufacturing Supply Chain Development for Modular Solar-Thermochemical Conversion Platform - CRADA 387 (Final Report)

June 2022

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Southern California Gas

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Pacific Northwest National Laboratory
Richland, Washington 99354

Cooperative Research and Development Agreement (CRADA) Final Report

Report Date: June 30, 2022

In accordance with Requirements set forth in the terms of the CRADA, this document is the CRADA Final Report, including a list of Subject Inventions, to be provided to PNNL Information Release who will forward to the DOE Office of Scientific and Technical Information as part of the commitment to the public to demonstrate results of federally funded research.

Parties to the Agreement:

Battelle Memorial Institute, Operator of Pacific Northwest National Laboratory

Oregon State University

STARS Technology Corporation

Southern California Gas Company

CRADA number: 387

CRADA Title: MANUFACTURING SUPPLY CHAIN DEVELOPMENT FOR MODULAR SOLAR-THERMOCHEMICAL CONVERSION PLATFORM

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DOE Program Office: Advanced Manufacturing Office, EERE

Joint Work Statement Funding Table showing DOE funding commitment:

	Federal		Cash in cost share		In kind cost share	
	PNNL	OSU	STARS	SoCalGas	STARS	OSU
Total	\$ 1,114,689	\$ 2,522,686	\$ 550,000	\$ 600,000	\$ 1,023,389	\$ 5,594,404

Executive Summary

Modular chemical process intensification (MCPI) is an emerging field where chemical processing is performed using small-scale modular equipment instead of conventional large centralized chemical plants. Conventional chemical plants benefit from economies of scale that encourage scale-up to ever larger plants. A goal of MCPI is to develop technology that intensifies processing so that equipment can be dramatically smaller and integrated into modular systems. Scale-up occurs by adding more modules in parallel rather than making the equipment larger. A key concept is that equipment and modules can ultimately be cheaper by leveraging economies of mass production, analogous to the automotive industry, in manufacturing the equipment. This project made significant progress toward this outcome by meeting the RAPID institute metric to reduce equipment cost by 20% for each doubling in manufacturing volume.

The MCPI application was thermochemical technology that is being commercialized by STARS Technology Corporation, one of the CRADA partners. The technology converts solar and renewable power to chemical energy to produce renewable hydrogen, fuels, and chemicals. The benefit to the public is reduction in greenhouse gases that are contributing to climate change.

The project transitioned the steam methane reforming (SMR) reactor from conventional fabrication methods to additive manufacturing (AM) direct metal laser sintering (DMLS) process. This is projected to reduce the cost of making a reactor by 58% when producing 100 reactors per year. Innovations in the DMLS process produced a patented design that reduces reactor weight by 60%. Reductions in material costs and processing time extend the DMLS advantage to higher production volumes. The new design promises to be 38% cheaper than the conventional processes at 1000 units per year. The resulting 87% reduction in the steam methane reforming (SMR) module cost in scaling from current costs meets the RAPID metric.

The project was successful in producing and testing the first ever additively manufactured SMR reactors. A reactor achieved over 82% efficiency in converting electric power to chemical energy, which is a world record for an inductively heated SMR. The project has contributed to the design and assembly of a first demonstration plant that is headed to a hydrogen bus filling station in Thousand Palms, CA.

Summary of Research Results

Project Activities

The project advanced equipment manufacturing methods to accelerate the commercial adoption of thermochemical technologies for converting solar energy and other heat sources into chemical energy and chemical products. The project was part of the “Modular Chemical Process Intensification Institute for Clean Energy Manufacturing”, also known as “Rapid Advancement in Process Intensification Deployment (RAPID)”, which was sponsored by DOE EERE’s Advanced Manufacturing Office. A project focus was to achieve the RAPID institute metric of reducing equipment cost by 20% for each doubling in manufacturing volume. Project outcomes resulted in 87% reduction in the steam methane reforming (SMR) module cost in scaling from 1 per annum to 512 from efficiencies of volume manufacturing, improvements in design for manufacturing, and manufacturing process development. Economies of mass production were projected from detailed process-based cost models that calculate costs of raw materials, capital equipment, facility, labor, maintenance, consumables, and utilities for each manufacturing process step.

The unique micro- and meso-channel components used in prior technology prototypes to date required specialty manufacturing approaches that are too expensive for the eventual commercial systems. The approach here was to reduce equipment costs by developing lower cost material systems, material savings, cheaper manufacturing processes, and novel equipment designs amenable to simpler and less expensive manufacturing processes. Oregon State University (OSU) focused on advancing additive manufacturing processes through inkjet technology, including developing the ability to ‘print’ an ink into a powder bed of a commercial direct metal laser sintering (DMLS) machine. The advancement enabled printing of lower-cost oxide dispersion strengthened (ODS) materials. A second advancement was to print enhanced thermal conductivity (ETC) structures within a microchannel device. OSU also developed components, including a high temperature recuperator, a water-gas shift reactor, and methanol and dimethyl ether synthesis reactors, using advanced manufacturing processes such as additive manufacturing (AM) and high-volume metal stamping.

PNNL focused primarily on the steam methane reforming reactor (SMR) component of the thermochemical conversion technology. The reactor is fed with steam and methane in a molar ratio of at least 3 which react at about 800°C to form a syngas stream containing hydrogen, carbon monoxide, carbon dioxide, and unreacted steam and methane. The endothermic reaction requires high temperature heat which is supplied conventionally by burning methane. The thermochemical technologies developed in this and prior projects use alternative heat sources such as concentrated solar heat and/or renewable electrical power. The product syngas has higher chemical energy than the methane feed, by up to 25%, so the process converts renewable energy to chemical energy that can be used to make power, fuel, or chemical products.

The baseline design of the SMR reactor at the start of the project is shown in Figure 1. The picture shows a prototype with heat exchangers attached to preheat incoming reactant streams. The side facing down in the picture on the right absorbs concentrated solar radiation that is used by the SMR reaction with over 70% solar to chemical energy conversion. The expensive high-temperature alloy components shown in the exploded assembly drawing in Figure 1 were previously fabricated using conventional machining, assembled by high temperature diffusion bonding, and finished using conventional welding and machining. The goal was to reduce cost through simpler and cheaper fabrication approaches.

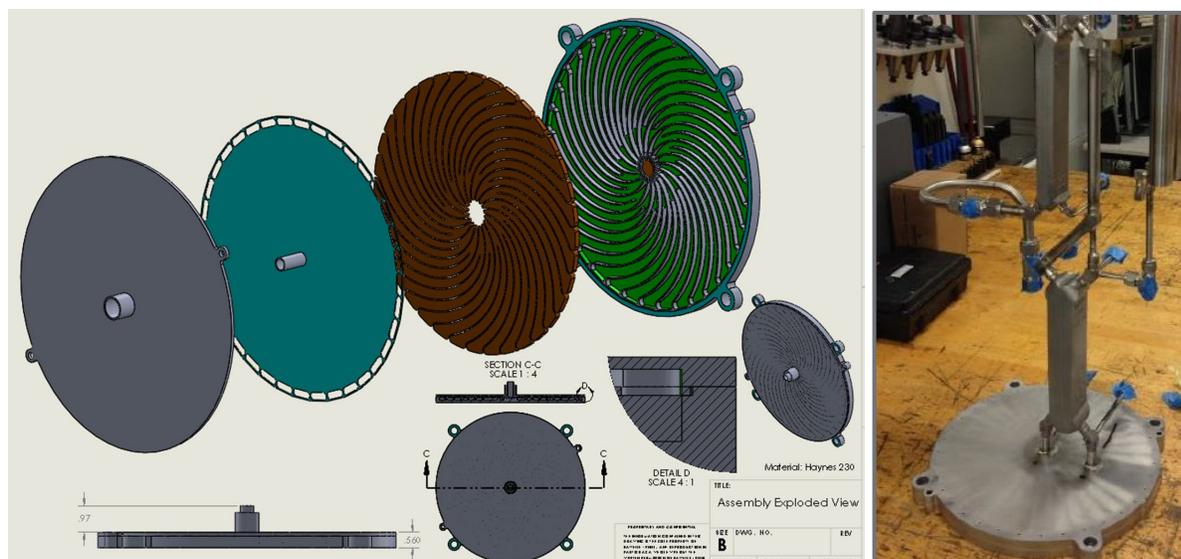


Figure 1. Baseline design of a solar-heated steam methane reforming chemical reactor.

This process intensification technology is inherently modular with a single system producing less than 100 kg/year of hydrogen. Scale-up is accomplished by adding parallel systems, referred to as numbering up. Economies of mass production of systems is the route to lowering costs, which are assessed by generating volume cost curves based on detailed analysis of the equipment manufacturing processes.

A detailed cost analysis of the reactor shown in Figure 1 is presented in Figure 2 assuming a new manufacturing facility with dedicated tooling. At relatively low volumes of reactors of 100 per year, the SMR reactor cost is estimated \$8800 with costs dominated by labor and tooling, the latter due to the \$2.7M capital investment. Raw material costs represent only about 8% of the cost and the remainder is split evenly between the fabrication steps. At higher production volumes of 1000 units per year, the cost drops to \$2400 per reactor primarily through better labor and tooling utilization, which shifts the breakdown toward raw materials and consumables. Machining high temperature alloys is expensive and poor material utilization of Haynes 230, priced at the time at \$56/kg, are the primary drivers.

The commercialization challenge becomes how to cross the 'manufacturing valley of death'. Early in the commercialization process when the demand is low, equipment costs can be prohibitive. The alternative is to identify fabrication techniques that are cost effective at low volumes, but that can be scaled with demand to be competitive at higher volumes. The process that was selected is the additive manufacturing process called direct metal laser sintering (DMLS). The process builds metal parts from a bed of metal powder using lasers to locally melt the powder one layer at a time, gradually building on previous layers until the entire structure is completed. DMLS metal powders are more expensive than bulk metals, which is offset by less waste with an additive process versus subtractive machining processes. In addition, additive manufacturing techniques allow for designs that cannot be built by conventional methods.

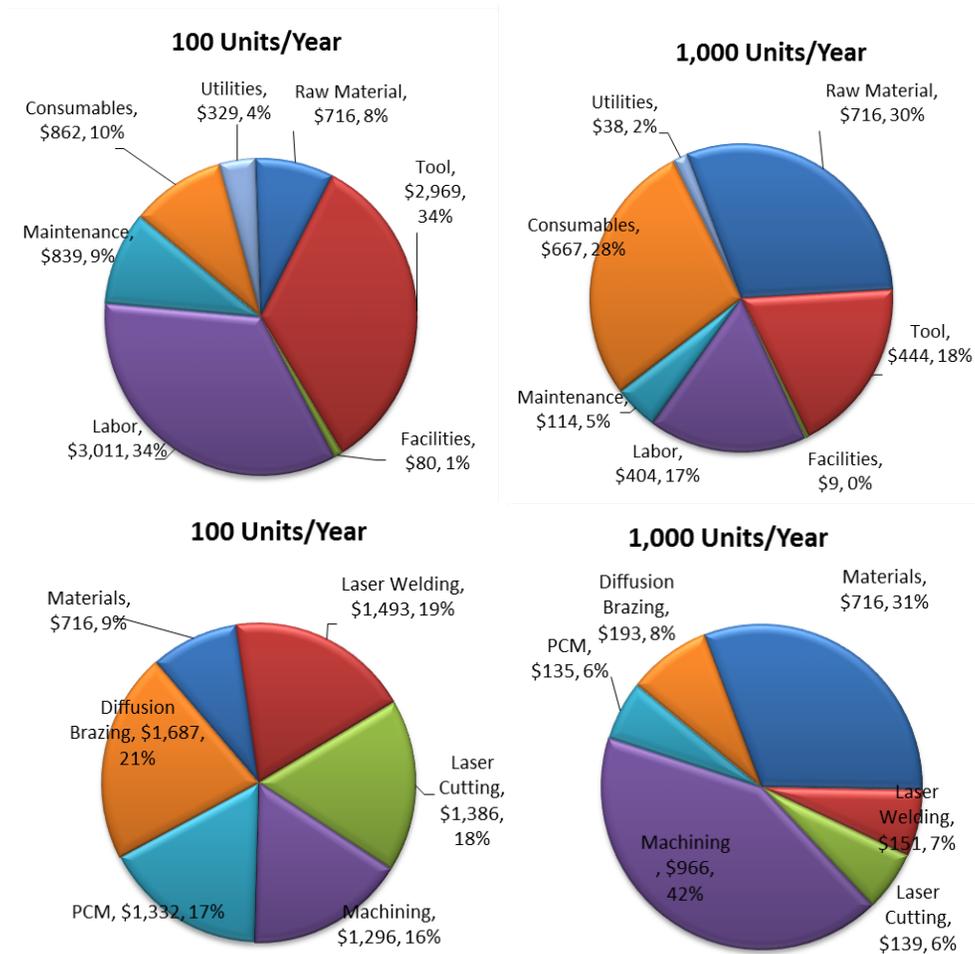


Figure 2. Cost breakdown of solar SMR reactor projected for 100 units/year and 1000 units/year.

The DMLS process can make metal parts that are fully dense and close to parent metal strength. However, the morphology and grain structure are significantly different than what is obtained from conventional material processing. Furthermore, the applications require thermal cycling, and the reactor is expected to last for 10 years with greater than 4000 thermal cycles. Test articles were fabricated, and a test stand constructed for thermal cycle testing. Figure 3 shows four test articles fabricated from the Haynes superalloy with both rectangular and circular flow channels and different wall thickness. The circular channel device with thinner walls was successfully thermally cycled for 4000 cycles between 300°C and 850°C by induction heating. Periodically, thermal cycling was stopped to visually inspect for deformation and to perform a vacuum leak check. The test device maintained hermeticity throughout the testing and showed minimal deformation. The device was then subjected to pressure testing at the end of the thermal cycling. The device was maintained at a temperature above 800°C while pressurizing the device in increments up to 100 psi, which is above the target SMR operating pressure of 5 bar. This task concluded that Haynes devices made by DMLS additive manufacturing have sufficient mechanical integrity for the intended applications and lifetime.

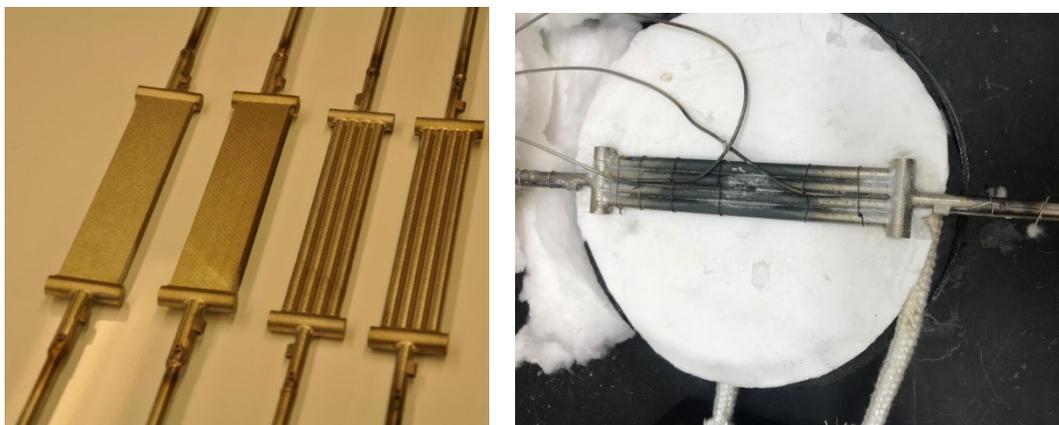


Figure 3. Thermal cycling of DMLS test articles.

Two designs for SMR reactor concepts were developed and patented. The first concept represented in Figure 4 is to fabricate the reactor in two pieces, a main body with openings for inserting catalyst structures into the reaction channels and an outer ring to cover the openings once catalyst is in place. The picture on the right in Figure 4 shows a 1/5-scale prototype of this reactor concept still inside the DMLS machine, which was the first ever additively manufactured SMR reactor. Cost modeling of this reactor concept indicated a manufacturing facility dedicated to fabricating this design could produce 100 units/year at \$3700 each, a 58% savings over the baseline stacked-plate design costing \$8800 each at 100 units/yr. At very high volumes, conventional fabrication is projected to be cheaper, and the breakeven point is projected at about 1000 units/yr. Consequently, this AM design concept represents substantial progress toward affordable manufacturing scale-up of the SMR reactors.



Figure 4. DMLS SMR reactor with catalyst inserted after fabrication.

The second concept is referred to as the embedded catalyst design, where the DMLS process is stopped and catalyst structures inserted into the reaction channels before continuing the DMLS process to complete the reactor. Consequently, the catalyst is entombed in the reactor during fabrication and no further manufacturing steps are required to complete a reactor. This concept enables flexibility to redesign the reactor for further cost savings. The conceptual drawing on the left in Figure 5 consists of bifurcated tubes that split periodically as they extend from the center of the cylindrical reactor to the perimeter. By taking advantage of hoop stresses

to contain the internal pressure, the walls can be considerably thinner, thereby requiring less material and less DMLS processing time to fabricate the reactor. Design calculations predict 60% savings in reactor weight. Comparing concepts at 1000 annual manufacturing volume, the conventionally machined concept costs \$2400 each, the AM inserted catalyst version in Figure 4 is \$2200 each, and the AM embedded catalyst concept is projected at \$1500 each. Consequently, this innovative concept, which depends on the design flexibility afforded by the DMLS process, can save 38% of equipment cost even at 1000 units per year or about 20 per week.

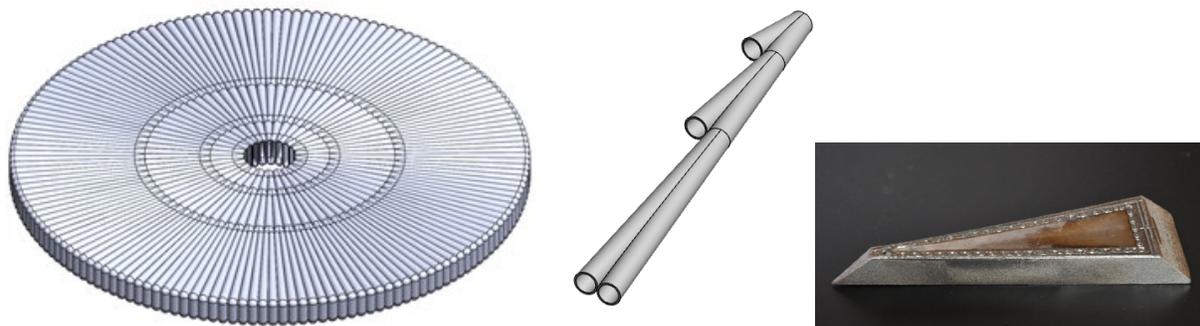


Figure 5. SMR reactor with catalyst embedded in the reactor during DMLS fabrication.

The embedded catalyst concept does require innovation in the DMLS fabrication process. The process must be stopped mid-run, powder removed from the reaction channels, catalyst structures inserted, and then the process resumed to complete the reactor. This process was demonstrated with a series of test articles and test runs performed with i3DMFG, a DMLS 3D printing vendor. The last tests were performed with the test article shown on the right in Figure 5. After the triangular piece was fabricated by DMLS, the machine was opened to remove powder from the cavity, and the metal lid was placed over the top. The DMLS laser was used to weld the lid on to prevent it from lifting off and disrupting subsequent DMLS processing. The success of these trials established confidence in fabricating a sub-scale test reactor, which was part of the project scope. However, completing full-scale reactors for deployment, as described below, took priority and progress in developing this concept was deferred.

The disk-style SMR reactors were originally designed and tested with concentrated solar heating using parabolic solar dish concentrators. The initial application targets were redirected to induction heating using renewable electrical power to give higher capacity factors and better economics. To leverage the TRL7 solar reactors, pancake style induction coils used in cooktop stoves were adapted, with the goal of using commercially available induction heating technology.

The first design of an induction heated SMR reactor is shown in Figure 6, which targeted 33 kg/day of hydrogen production requiring 15 kg/hr of feed at 3:1 steam to carbon ratio and 90% conversion at 800°C and 5 bar pressure. The desire for operating flexibility required a structural design point of 20 bar. Pressure and thermal stresses were assessed using COMSOL finite element analysis models. After importing the CAD drawing from SOLIDWORKS into COMSOL, the reacting flow model was used to predict temperature profiles, as illustrated in Figure 7. Temperature profiles were imported into a mechanical stress model to ensure the maximum thermal and pressure stresses were below the yield stress of the Haynes 282 alloy. Figure 8 illustrates a von Mises stress profile on the surface of the reactor. Several ideas for reducing stresses were developed and included in a provisional patent application.

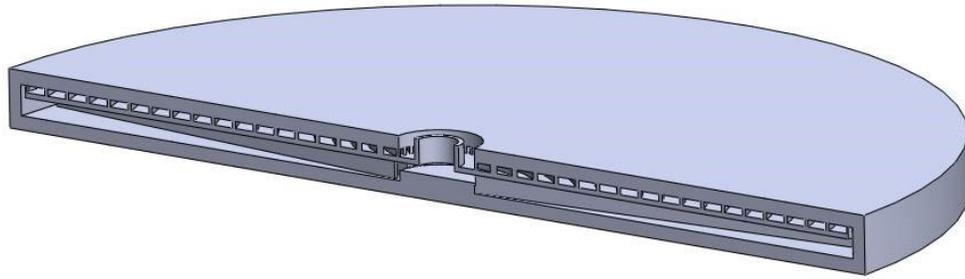


Figure 6. CAD drawing of a full-scale SMR reactor with cut-away on a centerline.

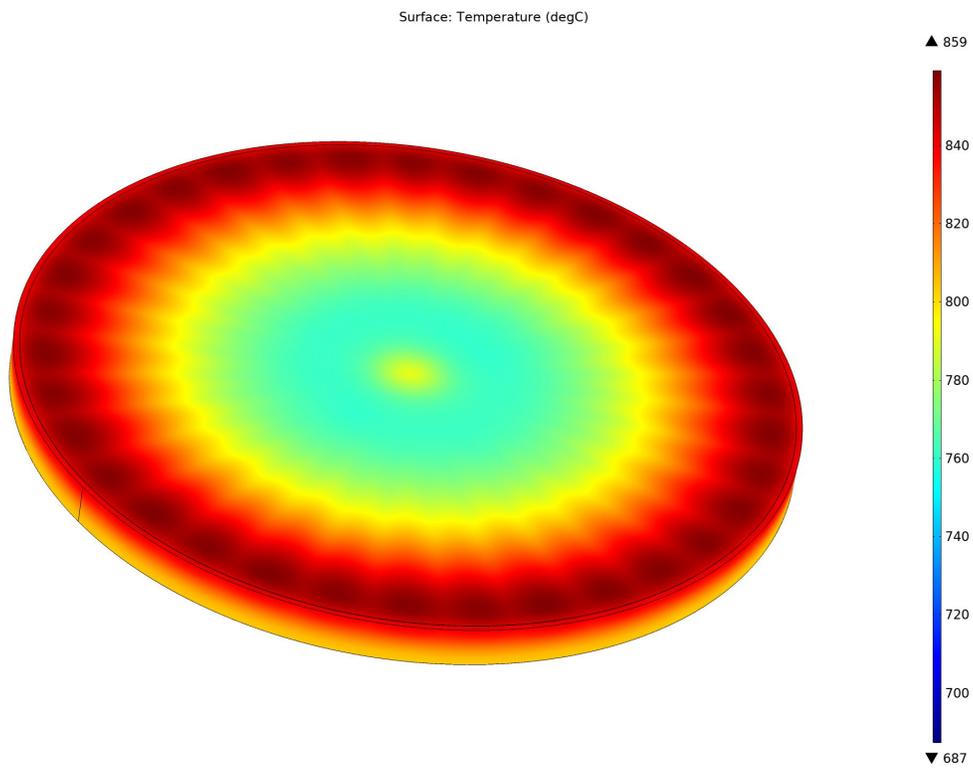


Figure 7. Temperature profile of the reactor face with heating uniformly distributed over surface.

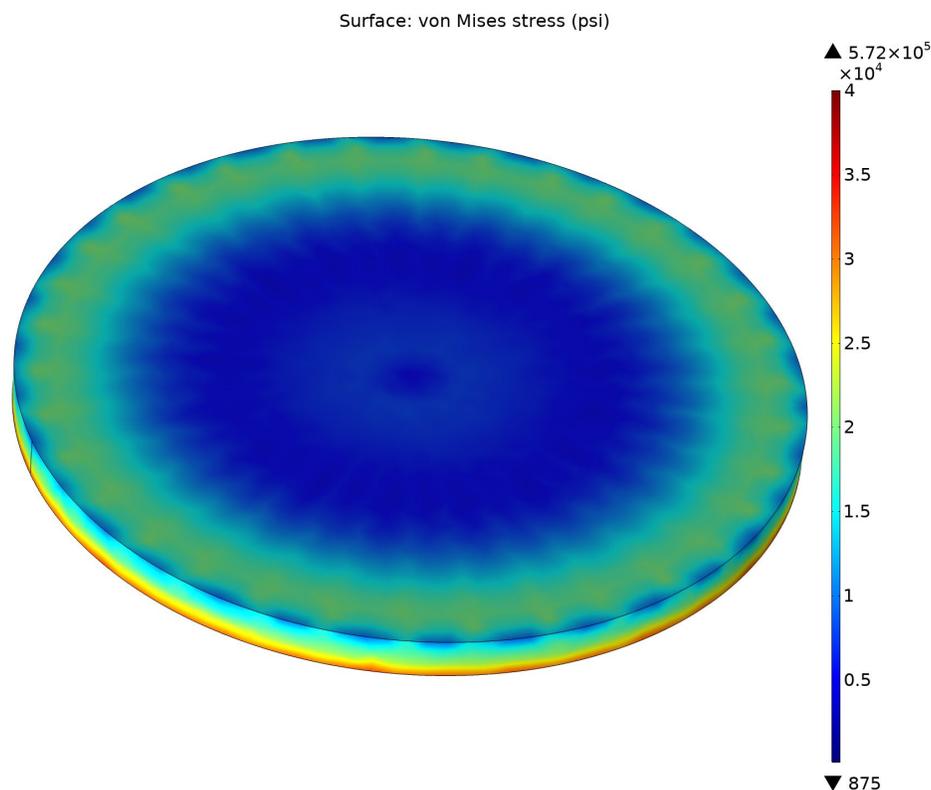


Figure 8. Stress profile of the reactor face with heating uniformly distributed over surface.

Development of an automated catalyst manufacturing capability was included in the project scope. Figure 9 shows a piece of catalyst-coated FeCrAlY foam being inserted into a reaction channel of an AM reactor. A total of 36 pieces were needed for the reactor in Figure 6. Previously, each piece was manually wash-coated multiple times and dried between cycles. This labor-intensive process represents significant manufacturing costs and is subject to variability between operators. The goal was to reduce cost and time for fabricating the catalysts for a reactor by 30% and 50%, respectively, through automation, while also improving reproducibility. Figure 10 shows the automated catalyst coating system developed by the project. The FeCrAlY catalyst pieces are placed on the tray of a circular food dryer. A gantry robot magnetically picks a catalyst piece from the tray and places it into the gray-colored bath. The bath then fills with the catalyst slurry to fill the porous substrate. After the bath is drained, an air knife passes over the catalyst pieces to remove excess liquid and prevent plugging of the pores. The robot returns the catalyst piece to its position on the drying tray, and the tray rotates to advance to the next piece. By the time the tray completes a full rotation, the catalyst is dry and ready for the next coating. The process continues until the catalyst pieces contain the target catalyst mass, which typically takes 6 repetitions. Coating a set of catalysts for a reactor now takes about 24 hours versus 5 days with the previous manual process. Labor savings are greater than 30% for the overall catalyst fabrication process.



Figure 9. Insertion of wash-coated FeCrAlY foam catalyst pieces into the full-scale AM SMR reactor.

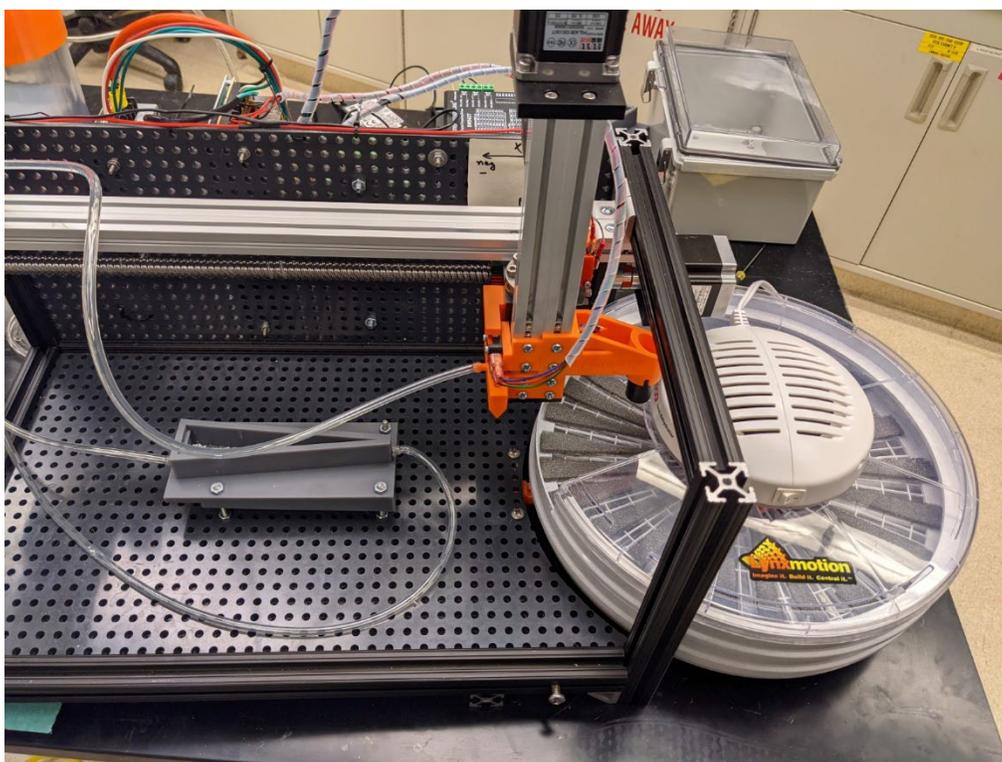


Figure 10. Automated catalyst coating system.

Two versions of full-scale SMR reactors were designed and constructed. The first was a two-layer reactor shown in Figure 6 and Figure 9, designed for 12 kW net heat input to produce 33 kg/day of hydrogen. Induction heating is applied to one side where reactants—methane and steam—flow radially through reaction channels containing catalysts from the center to the perimeter. The flow then passes through openings to the other layer at the perimeter and flows radially back to the central header where it exits the reactor. The return channels serve to recuperate heat back to the reaction channels to improve overall energy efficiency of the process.

The second design was a 3-layer reactor that is shown in Figure 11. The added layer contains a second set of reaction channels, and induction heating coils are placed on both sides of the reactor. A specially designed header, shown on the right of Figure 11, distributes flow uniformly to the top and bottom layers through the annulus of the header. Flow is transferred from the top and bottom layers to the middle layer at the reactor perimeter, which flows radially inward to the center pipe of the header. This design potentially doubles the productivity of a given reactor with a marginal increase in manufacturing cost. As with the 2-layer reactor, catalyst is inserted into openings in the top and bottom layers at the perimeter before having the ring installed and welded shut. Considerable effort was made analyzing this design using FEA models to ensure stresses did not exceed the material strength of Haynes 282. A picture of a 3-layer reactor made by i3DMFG using DMLS additive manufacturing is shown on the left in Figure 12. The outer ring was removed using wire EDM, and the next step is to insert the catalyst into the reaction channels. The right picture in Figure 12 shows an E.G.O. coil installed on the side where the high temperature recuperator is attached to the header. The 3-layer reactors are being used in the first STARS demonstration system being deployed to a California hydrogen filling station.

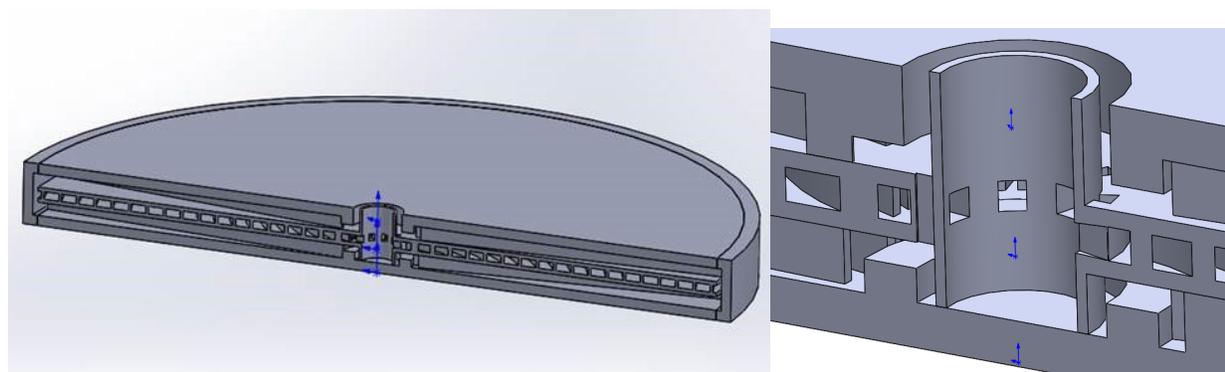


Figure 11. Three-layer inductively heated SMR reactor.



Figure 12. Three-layer inductively heated SMR reactor fabricated by i3DMFG.

A test bed was constructed to test components and subsystems of thermochemical conversion systems up to about 5 kg/day of hydrogen production. A process and instrumentation diagram (P&ID) of the system is shown in Figure 13. The test bed is capable of testing components and subsystems consisting of SMR reactors, heat exchangers, vaporizers, combustors, and downstream reactors including water-gas shift (WGS) and methanol and DME synthesis reactors. Bottled gas supplies allow for feeding of methane, natural gas, or mixed gases to simulate fuel mixes and reactor feed streams. Water will be metered to a vaporizer to produce steam and air supplied for combustion processes. A gas chromatograph will measure composition of dried product gases. A data acquisition and control system will collect temperatures and pressures and control feed flows, and will have additional i/o available for the specific test component or subsystem. Modifications from the P&ID shown in Figure 13 include adding an HPLC pump with a flow loop to feed the steam generator, heat exchangers to condense water from the effluent stream, and a second trap for collecting and removing liquid water.

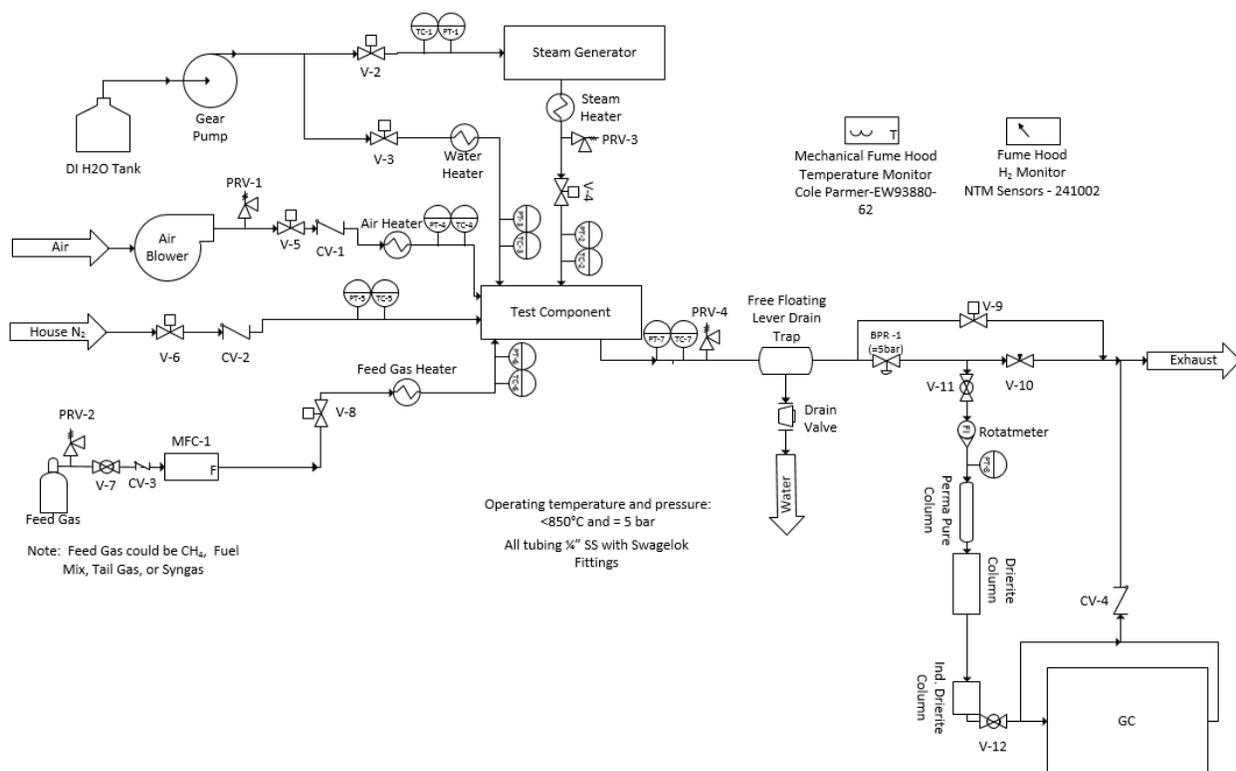


Figure 13. P&ID for the laboratory test bed for testing components and subsystems at the scale of 5 kg/day of hydrogen.

The test bed was used to test inductively heated 2-layer SMR reactors. The objective was to use off-the-shelf induction heating systems that have high reliability. Commercial E.G.O. cooktop technology was selected with options for up to 5 kW delivered power at 208 V or 8 kW at 400 V. A 5 kW, 208V system was adapted and integrated into the test bed and control system to modulate delivered power to the induction coils to control the average of 12 thermocouples on the perimeter of the reactor. The reactor with the higher temperature recuperator (HTR) on top is shown in Figure 14. The black square below the reactor is the E.G.O. induction coil. The insulated reactor with thermocouples installed on the perimeter is shown on the right of Figure 14. The HTR was insulated before testing.



Figure 14. Full-scale 2-layer reactor on top of a commercial E.G.O. induction coil (black square) on left and with insulation and perimeter thermocouples on the right.

Considerable efforts were made to improve the induction coupling between the E.G.O. system and the Haynes 282 reactor using Cobalt-Iron that has Curie temperature of 950°C. Maintaining adherence between the Cobalt-Iron sheets and the Haynes 282 reactors was problematic due to a significant mismatch in the coefficient of thermal expansion. In addition, the Cobalt-Iron material is susceptible to oxidative degradation at high temperatures. Early efforts to attach a cobalt-iron sheet using thermal paste resulted in delamination, as shown in Figure 15. Eventually, the cobalt-iron was segmented, coated, and brazed onto the SMR reactor as shown in Figure 16. Thermal cycle testing demonstrated adherence could be maintained and the Cobalt-Iron had significantly less oxidative degradation at high temperatures.



Figure 15. Delamination of the cobalt-iron plate from the bottom of the SMR reactor.



Figure 16. Segmented pieces of Cobalt-Iron coated and brazed to the top of a 3-layer 282 Haynes SMR reactor.

Progress in increasing productivity of the 2-layer SMR reactor is shown in Figure 17. The x-axis is the amount of power consumed by the induction heating system. The y-axis is the increase in energy content of the process stream, as indicated by the change in higher heating value. The ratio represents the energy efficiency of converting electrical power to chemical energy. The earlier runs suffered from poor inductive coupling which limited the power input to less than 3 kW. Higher power levels were successively achieved as the inductive coupling was improved. The 5-kW rated power was nearly attained with the final run that was performed in December 2021, using the Cobalt-Iron configuration shown in Figure 16.

Efficiency of converting electrical power to chemical energy is critical for reaching target costs for delivering hydrogen to fuel cell vehicle filling stations and other applications. As indicated by the labels in Figure 17, over 82% efficiency was achieved with the 2-layer reactor, which is a world record for an inductively heated SMR. However, future work will focus on increasing this efficiency to over 90% by reducing losses, including heat losses to ambient and ohmic losses in the induction coils. Continued development of the induction heating system by E.G.O. will facilitate higher heating powers, which will dilute the losses and enable higher efficiency. Higher power levels will also increase productivity of each reactor, thereby reducing capital expenditure (CAPEX).

The CRADA project has also assisted with building the first STARS demonstration system that will be deployed to a Sunline Transit Agency site in Thousand Palms, CA. PNNL has fabricated the catalysts and assisted with assembly of the three 2-layer SMR reactors that are installed in the demonstration system shown in Figure 18. The system was designed by Barr Engineering and constructed by HiLine Engineering and Fabrication, Inc. of Richland, WA, two of STARS commercialization partners. The goal is to produce 165 kg/day of hydrogen from the full system that will have a total of 6 three-layer reactors. In follow-on projects PNNL will continue to support the demonstration as well as continue technology development.

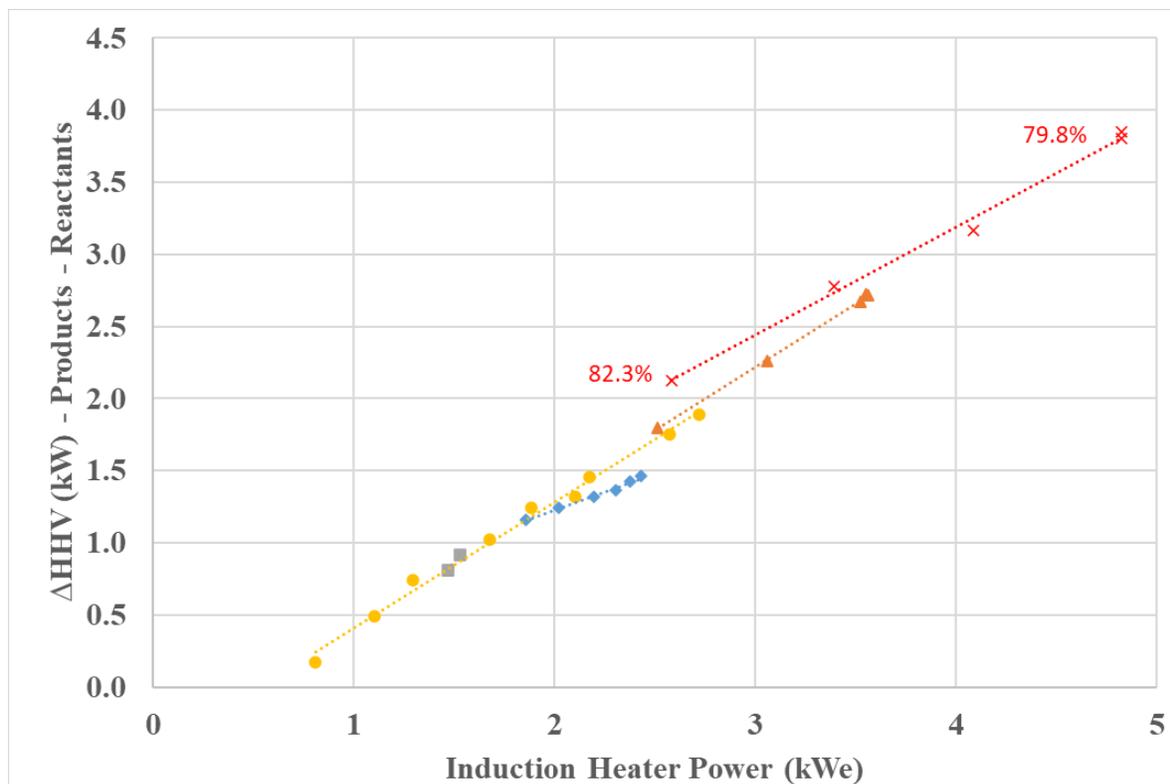


Figure 17. Change in higher heating value of the process stream versus induction power consumption (labels indicate efficiency) with the full-scale 2-layer SMR reactor with an E.G.O. induction heater for Run1-2/24/2021 (◆), Run 2-3/13/2021 (●), Run3-7/9/2024 (■), Run4-8/2/2021 (▲), and Run5-12/14/2021 (X).

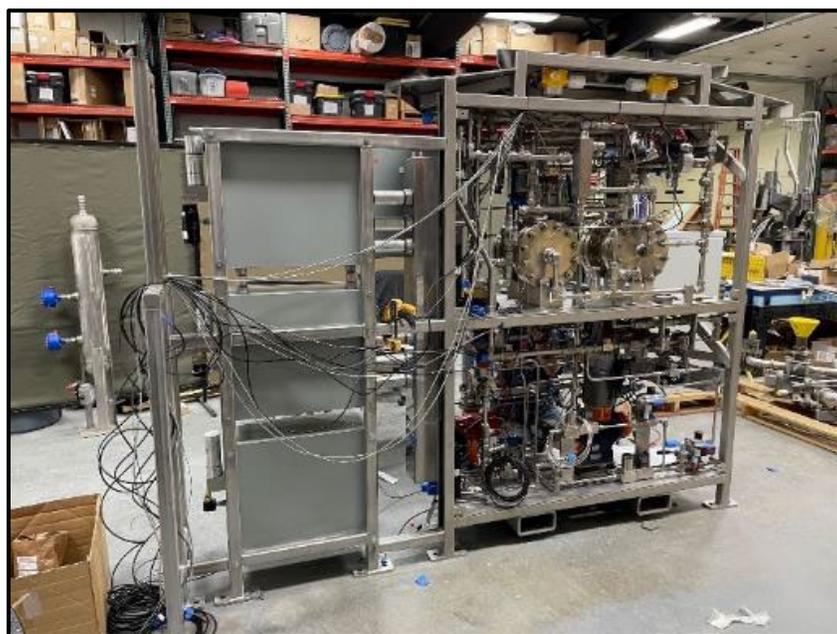


Figure 18. STARS 165 H₂ generator (Beta-1) in assembly at HiLine Engineering, Richland, WA.

Subject Inventions

1. TeGrotenhuis, WE, PH Humble, CK Clayton, TG Veldman, RS Wegeng, RF Zheng, “Enhanced microchannel or mesochannel devices and methods of additively manufacturing the same”, U.S. Patent 10,981,141, April 2021.
2. Inventors TBD, “Process Intensive Micro- and Meso-Channel Methane Reforming Reactors with Reduced Thermal Stress”, U.S. Provisional Patent Application, March 2022.
3. Wegeng, R.S. and D. Walters, “Method and Apparatus for Inductively Heating Micro- and Meso-channel Process Systems”, PCT Patent Application WO 2022/061041A3, September 2021.
4. Wegeng, R.S. and D. Walters, “Method and Apparatus for Inductively Heating Micro- and Meso-channel Process Systems”, U.S. Provisional Patent Application, March 2022.

Products develop and transfer activities

Products developed under the CRADA include three designs for DMLS additively manufactured SMR reactors, including a 1/5-scale 2-layer reactor, a full-scale 2-layer reactor, and a full-scale 3-layer reactor. Methodology for successfully printing reactors was developed with i3DMFG vendor and know-how was transferred to STARS Technology Corporation. PNNL developed methodology for coating and brazing Cobalt-Iron pieces to the surface of SMR reactors to enhance inductive coupling to commercial induction heating systems, which was also transferred to STARS. An automated catalyst manufacturing system was developed, and multiple batches of catalysts were fabricated and delivered to STARS for the first 6 full-scale 3-layer reactors. Technical assistance was provided to STARS during shakedown testing of the first demonstration system to be deployed in Thousand Palms, CA.

The project was successful in providing a route for STARS to cross the ‘manufacturing valley of death’ in commercializing the thermochemical technologies. PNNL successfully reduced the cost of solar and electrically SMR reactors by 58% at a manufacturing volume of 100 per year by transitioning from conventional fabrication methods to additive manufacturing. Further innovations promise to save another 38% at 1000 per year by leveraging AM design flexibility.

The project generated one issued U.S. patent with pending U.S. divisional and PCT applications, and a U.S. provisional patent application. STARS Technology Corporation filed a U.S. patent application and a PCT patent application, both which are under review for PNNL co-inventorship. See above patent list.

Presentations and publications:

1. BK Paul, S Pasebani, B Fronk, G Jovanovic, R Wegeng, W TeGrotenhuis, R Zheng and D Brown, “Manufacturing Supply Chain Development for Solar Thermochemical Modules”, presented at the AIChE Annual Meeting, Minneapolis, MN, October 2017.
2. BK Paul and W TeGrotenhuis, “Manufacturing Supply Chain Development for the STARS Technology Modular Solar-Thermochemical Conversion Platform”, presented at the AIChE Annual Meeting, New Orleans, LA, April 2019.

3. Zheng, RF, DR Bottenus, WE TeGrotenhuis, PS McNeff, P Humble, RS Wegeng, "Induction Heated Modular Reactor for High Efficiency Steam Methane Reforming and Hydrogen Production", manuscript in preparation.
4. McNeff, PS, DR Bottenus, WE TeGrotenhuis, P Humble, RS Wegeng, RF Zheng, "Additive Manufacturing of Compact Modular Steam Methane Reforming Reactor", manuscript in preparation.

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