DOE-ID NEPA CX DETERMINATION
Idaho National Laboratory

CX Posting No.: DOE-ID-INL-20-086

SECTION A. Project Title: Advanced Materials for CSP Molten Salt Storage – Heat Exchangers

SECTION B. Project Description and Purpose:

Powdermet, Inc. is a nationally recognized nanotechnology and advanced materials research and development (R&D) organization operating from a 54,000 sq. ft. manufacturing and R&D center located in Euclid, Ohio. Sponsor develops, matures, and transitions breakthrough materials innovations that enable reduced weight, resource consumption, environmental footprint and life-cycle costs, while also increasing energy-efficiency based value-creation, which is gained through engineered nano-scale features and hierarchically structures of metal and/or ceramic phases in a structure.

Powdermet was awarded a Department of Energy (DOE) Office of Science Phase II Small Business Innovation Research (SBIR) award for Funding Opportunity Number: DE-FOA-0002156 Topic 12f Solar. Powdermet reached out to Idaho National Laboratory (INL) to assist with process development of multifunctional thermal materials for molten salt thermal energy storage systems because contractor is currently performing research in the areas of advanced manufacturing and specifically spark plasma sintering (SPS).

INL is well known for R&D expertise within the advanced manufacturing space with access to equipment such as the Fuji Dr. Sinter SPS-515S with a 5kN load and 1800 amp capability, a custom Thermal Technology DCS-5 system with 10kN and 5000 amp capability, and a Thermal Technology DCS-800 with 800 ton and 150 kA capability, which are unavailable anywhere elsewhere.

The proposed work and technology require further development in order to overcome material, design, and manufacturing technical barriers, and the Powdermet recognizes INL's expertise and capabilities in this area.

Thermal energy storage is among the most scalable and efficient methods to store renewable energy such as concentrating solar power for grid levelling applications. This SBIR/STTR project will demonstrate suitable materials of construction that enable Gen 3, higher efficiency concentrating solar power thermal energy storage systems to be built reliably and cost effectively. The specific technical objectives of the Phase II DOE SBIR program are listed below:

1. Evaluate high thermal conductive additive fillers to down select to two primary Hybritherm metal matrix composite (MMC) formulations that meet technical and cost criteria.
2. Complete a round of processing optimization on primary candidate formulations then complete thermal-mechanical characterization for trade evaluations.
3. Complete a trade study on several heat exchanger core designs to down select to one formulation and one core design to move forward with in fabricating.
4. Complete a manufacturing investigation for selection of processing methods for component fabrication, and complete fabrication of a prototype heat exchanger core for testing under operating conditions.
5. Complete prototype heat exchanger core testing under operating conditions, demonstrating the operational value of Hybritherm MMC in power conversion under high temperature and corrosive conditions.

This program is specifically focused on validating cost-effective, multifunctional materials for CSP Gen 3 thermal energy storage systems, including high temperature heat exchangers capable of operating in the 700-800°C range.

Tasks

<u>Task 1. Material optimization and characterization.</u>

Starting the program off, Powdermet will complete a preliminary candidate screening regarding high thermal conductivity additives – tungsten carbide (WC), cubic boron nitride (cBN), silicon carbide (SiC), and diamond (Di). Phase I's hexagonal boron nitride trials demonstrated a loss of its ability to increase thermal conductivity at the application's high temperatures >500°C – it is believed the hexagonal boron nitride's crystal structure which possesses low thermal conductivity in the c-direction, k<3W/mK, contributed to the rapid k decline in the Phase I's Hyberitherm MMC. Switching the thermal conductive additive to a higher k value material will provide the desired results at the higher temperature range.

The investigation will address potential concerns for chemical reactivity and phase changes that could occur with the additives and the metal matrix during sintering. Chemical reactivity of additives to molten salt and CO_2 are less of a concern as published work reports little to no reaction at temperature below 1,000°C, and additives will possess a protective nickel coating. If unwanted additive matrix reactions are encountered, Powdermet is prepared to apply any one of its powder-particle coating technologies – For example, if UNS-R30783 with diamond is deemed a desirable candidate and pressure or temperature conditions results in unacceptable chemical interactions a SiO_2 coating can be applied (100nm to several µm) to inhibit the undesirable interaction at processing conditions. Such decisions are scheduled to be resolved early within the first 3 months of the program.

Powdermet will continue to use its Phase I selected alloys UNS-R30783 (comparable to Special Metal's Inconel 783) and UNS-N10003 (comparable to Haynes Hastelloy-N) and Powdermet's Hybritherm™ WC MMC alloy formulation. These alloy compositions have demonstrated low susceptibility to molten salt and supercritical CO_2 corrosion. There has been a noticed susceptibility of high chromium content (>8% weight) alloys to ICL molten salts at the higher temperatures >650°C – the chromium is observed being leached from these alloys. All alloy choices have <8% chromium content. R30783 possesses near similar properties to, IN909 but with improved oxidation resistance, especially regarding stress assisted grain boundary oxidation. N10003 was a recommended alloy showing promising performance over Haynes 230 when comparing molten salt corrosion resistance.

The alloy composites with high thermal conductive additives (a reduced matrix of all options will be evaluated) will be mixed in several loadings to produce 1" pucks (10-30 formulations) and trialed through spark plasma sintering (SPS) processing at CalNano. Samples will be microscopically analyzed in house at Powdermet and thermal conductivity (ASTM E1461) measured through Orton Ceramic's testing facilities. Microscopic analysis will evaluate the compatibility between the additives, coatings, and metal matrix. Thermal conductivity tests will be taken up through 800°C to measure the MMCs' thermal characteristics at higher temperature and confirm theoretical predictions. The thermal properties along with in-house comparative mechanical property testing (tensile, flexural, and/or compression) on the various loading amounts will define a set of performance windows based on MMC loading, and lead to a down selection. Selection of two candidate Hybritherm MMC materials will Thermal, mechanical, and corrosion properties of candidate materials will be used in evaluating the overall performance of Powdermet's materials. A down-selection to one Hybritherm MMC material will be used to proceed through the rest of the program.

Task 2. Processing optimization and expansive property testing.

Powdermet will process another ~15kg of primary candidate material for SPS billet consolidation at INL's spark plasma sintering facilities. Three billets plates 8"⌀ x 0.5" thick will be pressed. Enough material will be provided to allow INL to gain more familiarity with the Hybrimet MMC materials. Prior sintering results will be used to develop and hone in process sintering parameters to optimize the parts final properties. INL will develop sintering process parameters:

1) A target sintering temperature and pressure appropriate to the material will be selected based upon prior work by Powdermet. The maximum sintering temperature is ~90% of the melting temperature of the lowest melting temperature material in the samples. With guidance from Powdermet on any necessary lower temperature bake outs/holds, INL will then apply heating rates of between 100-400 C/min and a target hold time of 5-20 minutes.

2) Using a room temperature equilibrated SPS machine, the sintering run will be conducted, and the sample will remain in the SPS equipment until the following day, and the final sample height determined in comparison to the green dense sample height. Powdermet will be contacted to determine whether full density has been achieved. If so, the experiments will continue as described below. If not, the initial sintering parameters will be adjusted in coordination with Powdermet.

3) The fully dense specimen will again have the same process parameters applied as during sintering in Steps 1-4. The resulting shrinkage/expansion data will then be removed from the data from Step 3, and will provide a true densification curve for that material.

4) From the densification curve, the rate of shrinkage will be determined to find the maximum shrinkage rate and the temperature (Tshrink) at which this occurs. If the curve is smooth and not discontinuous, then the following steps can guide the reduction in necessary sintering temperature.

5) INL will select a sintering temperature between Tshrink and the temperature used in Steps 3 and 4, and will calculate a hold time based upon an equivalent mass flow (shrinkage) at the lower temperature. The density of the samples from Steps 3 and 6 will be compared by Powdermet. Based on the results, the following 3 to 7 samples tested will be incrementally increased in density.

6) INL will produce densification curves, based upon the best case processes from Step (6), for samples having final sintered thicknesses as needed by Powdermet.

The key deliverables here are densified specimens (up to 15 total), with diameters less than or equal to 50mm), process parameters associated with specimens, and the densification curves associated with the material of interest and the geometry. Processed plates will be EDM machined for coupon samples to conduct a more extensive round of thermal and mechanical property testing.

RT Tensile (ASTM E8): 3 samples

RT compression (ASTM E9): 3 samples

RT flexure strength *ASTM C1161: 3 samples

750°C Tensile* (ASTM E21): 3 samples

750°C Compression (ASTM E9):3 samples

CTE (ASTM E228, RT-1000C): 3 samples

Thermal conductivity (ASTM E1461) (RT-750C): 3 samples

*or equivalent testing

In addition, Powdermet will fabricate 5-15 creep/burst specimens. These creep coupons will be a 5-channel sandwich design diffusion bonded together. Brayton Energy will conduct the creep-burst testing. This creep testing will contribute to providing confidence that Powdermet's HybriTherm MMC materials can survive for its operational life. It will also provide valuable information in developing the diffusion bonding processing parameters for optimal bonding. It ultimately depends on the core design of the heat exchanger, but the failure mode will either be pure creep or creep-fatigue. Brayton's creep tests will place the samples under pure creep at high temperature until failure is reached. Comparison of the actual failure time to the predicted failure time will be used to quantify the performance. Creep testing is planned to run between 50-2,000 hours under 3,000psi at 650-750°C with industrial grade CO_2. Dimensions and photographs will document the start and test progress to specimen failure. Several failed samples will be cut, polished, and microscopically analyzed at the failure segment, and around diffusion bonded regions if they survive. Brayton will use the testing data to create an ANSYS model of the material to predict life and failure points, and this will be used in determining sample temperature and pressure limits – providing guidelines for HX core design. Average property values will then be used in identifying a heat exchanger core design to move forward with in fabricating for prototype demonstration evaluations.

Task 3. Heat Exchanger core design

Material properties will be used in designing a heat exchanger (HX) core. University of Wisconsin-Madison and Brayton Energy will conduct a trade study evaluating designs that benefit from HybriTherm MMC's properties and are compatible with available manufacturing processes. This will consist of evaluation of whether wire EDM methods will be used to make through plate channels or a more scalable plunge EDM machining technique will be used to make the heat exchanger channels in the plates prior to diffusion bonding. Detailed ANSYS, CFD and heat transfer models will assess the thermal hydraulic and mechanical performance of the channel and header geometry with respect to performance, manufacturability and cost. Execution of analytical and computational design efforts will reduce key risks associated with the HX module trial. A preliminary example design, with a simple flow topology. Univ. Wisc. will complete a trade study and model the performance of Powdermet's HybriTherm MMC HX core against current SOA HX cores. The study will predict commercial unit size and related performance value. Powdermet will work in tandem providing the associative manufacturing cost.

Task 4. Manufacture of subscale components and HX core

Powdermet will process and provide additional primary candidate HybriTherm material for SPS consolidation at INL by SPS pressing of 3-5 billets plates 8"⌀ x 0.5" thick. Process parameters will have been evaluated and dialed in for optimal processing. Small 1"⌀ x 0.5" thick samples will be EDM machined and polished for diffusion bonding development by spark plasma bonding at INL. The diffusion bonding development will consist of the following steps:

1. INL will establish the necessary baseline (zero current, hot-pressing analogue) process variables and conditions. A limited parametric study will be performed in order to optimize the parameters to form diffusion welds comprised of two interfaces (3 sheets/plates). A 20 mm sheet diameter will most likely be used as this dimension is capable of being diffusion welded using currently available small scale SPS devices at INL. The primary independent variables to be determined in this phase are temperature, time, and pressure. Some secondary variables that may be considered are the surface finish of the plates to be diffusion weld and the possible use of an interlayer material (as/if determined appropriate by Powdermet). INL will fabricate ten diffusion weld plates in this phase. The interfaces will be characterized using scanning electron microscopy (SEM) and EBSD at Powdermet. Grain growth and pores will be evaluated to identify the optimal fabrication parameters. Ideally, diffusion welds interfaces will be indistinguishable from the base material as a result of grain growth across the interface. Therefore, the parameters chosen to be applied in the next step will meet the following conditions: 1) free of cracks, 2) result in the most grain growth across the interface, and 3) result in little to no pores at the interface. These chosen parameters will act as control variables in the next steps.

2. This step will fabricate diffusion welds comprised of two interfaces at two increasingly high current densities, using the configurations determined in step1. The primary variable will be the applied current density, possibly including variations in current density during processing. An additional variable that may be considered in this step is the impact of the vacuum quality. The characterization applied in Step 1, will also be applied in this step in order to down select to the most promising parameters to move forward. The same criteria used in Step 1 will be used for the down selection. The current intent is to fabricate and characterize ten diffusion-weld plates in this step.

3. The critical step here is to determine the role of increasing numbers of interfaces on the diffusion bonding efficacy and quality. Since each interface represents a given voltage drop, it is expected that there will be a difference in behavior moving from the 2 interfaces (3 plates) in Steps 1 and 2 to a larger number appropriate to a printed circuit heat exchanger design. INL will use the best parameters determined in Step 2 to bond samples with 5 interfaces, 10 interfaces, and 20 interfaces for Powdermet to characterize. If significant variations occur, further work outside the scope of this project will likely be necessary.

Diffusion bonding work will be trialed and developed with earlier billets and bonded pieces EDM machined for tensile or flexural bar testing. Spark plasma diffusion bonding has been successfully demonstrated previously for Powdermet's HybriTherm WC MMC composites. From pressed plates Powdermet will EDM machine (possibly plunge EDM with INL if needed) +2 multi-plate (7-8" ⌀) flow grids from pressed plates for its laboratory HX core prototype. These machined plates will be SPS diffusion bonded at INL to construct the prototype HX core.

Task 5. Complete laboratory testing on prototype HybriTherm MMC HX core

University of Wisconsin will complete a molten salt or sodium – to scCO2 demonstration trial with the fabricated prototype HX core. First the core will undergo helium leak testing followed by pressure testing to ensure structural integrity. The heat exchanger will then be tested at room temperature with a water loop to measure the associated pressure drop on either side. Following this high temperature prototypic testing will be conducted. This testing will be accomplished with an existing high pressure sCO2 pump cart and molten salt loop. The performance (heat transfer rate, pressure drop and overall effectiveness) of the heat exchange between sCO2 and molten will be measured and compared against the generated design models. A series of steady state and transient tests will be conducted to assess the performance and ramp rates. During the testing procedure the HX will be occasionally removed from service and undergo non-destructive X-ray CT analysis to evaluate the structural integrity of the heat exchanger. Efficiency in heat transfer and related corresponding overall power cycle efficiency will be reported on. The trial will demonstrate operation while contributing to further understanding future optimal fluid flow distribution efficiencies by pressure losses, flow, heat transfer, etc. The results will provide a demonstrative operation for Powdermet's HybriTherm MMC for HX core designs – thus bringing it up to a TRL 6. Performance values will aid in a commercialization assessment for defining market targets, and HX core design expectations (i.e., size, pressure and temperatures of optimal operation).

Additionally, under the commercialization maturation for HybriTherm MMC as a HX material, Powdermet will have solidified a supply chain and manufacturing up-stream set of facilities and collaborators for Phase III manufacturing.

Task 6. Commercialization

Powdermet with program partners will continue to actively refine the commercialization efforts that can include redefining the target market, size, and expectations. Powdermet will actively keep defining the organization of the manufacturing supply chain, facilities, and its collaborators for HX core production. Additionally, work will pursue the efforts to meet ASME BPVC Section VIII certification for compact HX. Commercialization efforts will be conducted in parallel to technical development efforts throughout the course of the program, with the goal of identifying early adopter customers for the Phase III maturation stage.

Task 7. Reporting

Contributing program participants will meet at least bi-monthly to report program tasks progress, provide project advice and coordinate progress. All contributing members will provide Powdermet bi-monthly reports on progress, and Powdermet will prepare full program reports as requested and final report.

INL's portion of the work will initially start at the INL Research Center using the smaller SPS units and then will proceed to the Energy Storage Laboratory using the 800 ton unit. The dies will either be returned to Powdermet or INL will keep them.

SECTION C. Environmental Aspects or Potential Sources of Impact:

Air Emissions

The SPS is operated under vacuum and the vacuum pump is exhausted through the building exhaust system.

Discharging to Surface-, Storm-, or Ground Water

N/A

Disturbing Cultural or Biological Resources

N/A

Generating and Managing Waste

Wipes with ethanol will be generated. Vacuum pump filters and oil traps may also be generated.

Releasing Contaminants

When chemicals are used, there is the potential to spill to air, water, or soil.

Using, Reusing, and Conserving Natural Resources

Material will be recycled to the extent practicable. All applicable waste will be diverted from disposal in the landfill when possible. Project personnel will use every opportunity to recycle, reuse, and recover materials and divert waste from the landfill when possible. The project will practice sustainable acquisition, as appropriate and practicable, by procuring construction materials that are energy efficient, water efficient, are bio-based in content, environmentally preferable, non-ozone depleting, have recycled content, and are non-toxic or less-toxic alternatives. New equipment will meet either the Energy Star or SNAP requirements as appropriate (see http://www.sftool.gov/GreenProcurement/).

SECTION D. Determine Recommended Level of Environmental Review, Identify Reference(s), and State Justification: Identify the applicable categorical exclusion from 10 Code of Federal Regulation (CFR) 1021, Appendix B, give the appropriate justification, and the approval date.

For Categorical Exclusions (CXs), the proposed action must not: (1) threaten a violation of applicable statutory, regulatory, or permit requirements for environmental, safety, and health, or similar requirements of Department of Energy (DOE) or Executive Orders; (2) require siting and construction or major expansion of waste storage, disposal, recovery, or treatment or facilities; (3) disturb hazardous substances, pollutants, contaminants, or Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)-excluded petroleum and natural gas products that pre-exist in the environment such that there would be uncontrolled or unpermitted releases; (4) have the potential to cause significant impacts on environmentally sensitive resources (see 10 CFR 1021). In addition, no extraordinary circumstances related to the proposal exist that would affect the significance of the action. In addition, the action is not "connected" to other action actions (40 CFR 1508.25(a)(1) and is not related to other actions with individually insignificant but cumulatively significant impacts (40 CFR 1608.27(b)(7)).

References:
10 CFR 1021, Appendix B to subpart D, items B3.6, "Small-scale research and development, laboratory operations, and pilot projects" and B1.31, "Installation or relocation of machinery and equipment."

Justification:
Project activities are consistent with 10 CFR 1021, Appendix B, B3.6, "Siting, construction, modification, operation, and decommissioning of facilities for small-scale research and development projects; conventional laboratory operations (such as preparation of chemical standards and sample analysis); and small-scale pilot projects (generally less than 2 years) frequently conducted to verify a concept before demonstration actions provided that construction or modification would be within or contiguous to a previously disturbed or developed area (where active utilities and currently used roads are readily accessible). Not included in this category are demonstration actions, meaning actions that are undertaken at a scale to show whether a technology would be viable on a larger scale and suitable for commercial deployment."

B1.31, "Installation or relocation and operation of machinery and equipment (including, but not limited to, laboratory equipment, electronic hardware, manufacturing machinery, maintenance equipment, and health and safety equipment), provided that uses of the installed or relocated items are consistent with the general missions of the receiving structure. Covered actions include modifications to an existing building, within or contiguous to a previously disturbed or developed area, that are necessary for equipment installation and relocation. Such modifications would not appreciably increase the footprint or height of the existing building or have the potential to cause significant changes to the type and magnitude of environmental impacts."

Is the project funded by the American Recovery and Reinvestment Act of 2009 (Recovery Act) ☐ Yes ☒ No

Approved by Jason Sturm, DOE-ID NEPA Compliance Officer on: 12/17/2020