High Efficiency Latent Heat Based Thermal Energy Storage System Compatible with Supercritical CO₂ Power Cycle

Applied Materials Division

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Executive Summary

As part of the SunShot Initiative's 2013 Laboratory Proposal Development program, Argonne National Laboratory (Argonne) developed and demonstrated a latent-heat thermal energy storage (LHTES) system using MgCl₂ ($T_{m.p.} \sim 714^{\circ}\text{C}$) phase change material (PCM) and graphite foam at the laboratory prototype-scale level. The current project focused on issues related to the fabrication and thermal performance feasibility of the LHTES module compatible with the supercritical CO₂ (s-CO₂) power cycle for concentrated solar power (CSP) applications. In collaboration with an industrial partner, several essential tasks were accomplished:

- (a) *Thermal analysis of a full-scale modular LHTES system.* Thermal simulations were conducted to ensure the compatibility with and feasibility of the LHTES in a s-CO₂ power cycle. A modular system was considered from the standpoint of ease of manufacturing and installation. Simulations showed that the use of graphite foam enhances the thermal performance of the PCM to meet the thermal storage system requirements.
- (b) *Investigation of the effects of eliminating brazing between the heat transfer fluid (HTF) pipe and graphite foam.* Gaps exist between the graphite foam–PCM combination and the HTF pipe. These gaps can adversely influence the heat transfer between the storage medium and the charging/discharging HTF. While brazing can eliminate these gaps, it increases the manufacturing cost. This effort focused on experimentally evaluating the effects of gap on the thermal performance of the LHTES system and showed that the brazing step could be eliminated.
- (c) *Demonstration of the use of low-cost alloys for the LHTES system.* This task demonstrated the viability of using low-cost inexpensive alloys such as stainless steel as the containment material, which leads to a substantial reduction in storage costs. This was established by conducting corrosion tests on various alloys (type 304 stainless steel, Inconel 625, and Haynes 230) in a realistic internal environment of the storage system. A process for PCM purification was developed and implemented. It was found that the corrosion rates/depths of the metal component materials decreased with exposure time. This observation suggests that since the LHTES is hermetically sealed and has limited corrosion-facilitating species, such as moisture and oxygen, with time these will be depleted and corrosion will be mitigated.
- (a) *Scale-up of the graphite foam process.* To address the need for large-scale foam production for a full-scale CSP plant, Argonne's commercial partner, Touchstone Research Laboratory (Touchstone), scaled up the foam production. Scaled-up material was characterized for thermo-physical properties and compared to the foam produced earlier at a small scale to validate the scaled-up production.
- (d) *Techno-economic analysis of full-scale modular LHTES systems.* This effort provided a complete techno-economic analysis on all aspects related to applications of the full-scale modular LHTES technology for CSP plants. Such factors included overall thermal performance, modular component parametric effects, energetic and exergy efficiencies, modular system cost, and so on. The results established a techno-economic basis for the viability of the modular LHTES technology for CSP plant applications.

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1. Background

As part of the SunShot Initiative's 2013 Laboratory Proposal Development Program (LPDP), Argonne National Laboratory (Argonne) has developed and demonstrated a latent-heat thermal energy storage (LHTES) system using magnesium chloride (MgCl₂) (phase change material [PCM], melting temperature of 714°C) and graphite foam at the laboratory prototype-scale level [1]. As part of this work, commercially available graphite foams, with varying porosities, were infiltrated by the MgCl₂ PCM to almost full capacity. Various system development efforts, such as brazing of components of the LHTES system, coating of the graphite foam, and testing of the corrosion behavior of the graphite foam and metal alloys, were carried out to build a working prototype. In addition, a laboratory test loop system was designed and built to conduct charging and discharging experiments. Charging and discharging tests showed that the performance of the laboratory prototype is close to that of the simulations. The experimental data and modeling showed the full-scale performance of the LHTES system met the SunShot Initiative's targets. Furthermore, a preliminary cost analysis was carried out. The focus of the system developed was to meet the heat transfer fluid (HTF) requirements for a steam Rankine power cycle; that is, the outlet HTF temperature from the storage system is expected to be >600–650°C.

As part of the current Sunshot Apollo project (the U.S. Department of Energy Office of Energy Efficiency & Renewable Energy's Solar Energy Technology Office), the overarching goal was to address key materials and system issues to further the LHTES technology for commercialization. The target requirement for the LHTES system was integration with the supercritical carbon dioxide (s-CO₂) power cycle to deliver an s-CO₂ outlet temperature of ~700°C. To accomplish this, the choice for the PCM was either MgCl₂ or a higher-melting-temperature PCM (sodium chloride [(NaCl)] with a melting point of 800°C and a latent heat of 481 kJ/kg [2]). Finally, since the cost of the full-scale system needed to meet SunShot's target of <\$15/kW_{th}, the focus was also on reducing the cost of the LHTES materials and components without affecting performance.

The work scope included the following key tasks:

- (1) Develop simulation models to predict the thermal performance of the full-scale LHTES system for the concentrating solar power (CSP) plant;
- (2) Conduct a parametric study of the LHTES system performance to optimize the LHTES system design;
- (3) Experimentally demonstrate the elimination of brazing between HTF pipe and graphite foam without adversely affecting thermal performance;
- (4) Conduct a feasibility study of using low-cost, "inexpensive" alloys for the LHTES containment system;
- (5) Scale up the graphite foam fabrication process;
- (6) Conduct a detailed techno-economic analysis of the full-scale LHTES system; and
- (7) In collaboration with the industrial partner, develop technology-to-market and commercialization plans for the LHTES technology.

2. Analysis of Full-Scale Modular LHTES Systems

2.1. Modular System Design

The proposed LHTES system uses a modular design. A modular LHTES system comprises many individual modules consisting of one or more HTF pipes surrounded by graphite foam infiltrated with PCM and contained in a second sealed pipe. Compared to the single-tank system, the modular system has several advantages:

- (a) *Ease of fabrication.* Modules can be fabricated in a factory under a controlled environment.
- (b) *Minimal on-site work.* The main work required on-site is to place the prefabricated modules into a support structure, connect them to HTF headers under the modules, and thermally insulate them at the outer boundaries of the modular system;
- (c) *Ease of maintenance.* A module can easily be repaired by disconnecting it from the heads and removing it from the top using a crane, and the system can continue to operate while the removed module is repaired through either replacement of the module or capping of the module connectors.
- (d) *Flexibility of design.* An LHTES modular system can be made up of any number of modules offering flexibility in matching a system to plant size.
- (e) *Cost-effectiveness.* The large-diameter thick-walled tank in the conventional system can be replaced with small-diameter thin-walled containment pipes resulting in cost savings.

For the LHTES system of this project, the design being considered is the modular system with seven HTF pipes, as shown in Figure 2-1. Figure 2-1a shows the individual module with seven HTF pipes; Figure 2-1b shows the overall design of the module; and Figure 2-1c shows several individual modules stacked together for a full storage system. This design was used in the thermal performance and cost analyses of the LHTES system. Further, for comparison purposes, a single-tank design was also considered, as shown in Figure 2-2.

Figure 2-1. Schematic of the LHTES system

Figure 2-2. Single-tank LHTES design

2.2. Technical Approach

To achieve the project goals, both modeling and experimental approaches were used. Thermal simulations and cost analyses were conducted to establish the benefits of using high-thermal-conductivity graphite foams in conjunction with PCMs and to develop designs for laboratory-scale prototypes. The step-by-step procedure for thermal simulations and cost analyses is schematically illustrated in Figure 2-3. The design and fabrication of the subsystem prototype were based on the numerical simulation results and the results and experience gained from the previous SunShot project. Based on the critical issues identified in the previous SunShot project, corresponding considerations on

many aspects related to the prototype, such as materials, dimensions, brazing/joining techniques, sealing methods, and so on, were taken into account.

Figure 2-3. Thermal simulation and cost analysis flowchart

2.3. System Thermal Performance

2.3.1. Basic Assumptions

The CSP plant capacity was assumed to be 100 MW_e with an 8-hour charging time and a 12-hour discharging time. Based on the efficiency of the s-CO₂ power cycle of approximately 50% [3], the LHTES system requires a thermal energy storage capacity of 200 MW_t (2400 MWh), which is equivalent to approximately 15,342 tons of the graphite foam–NaCl combination as the storage medium. The HTF pipes are inserted in the graphite foam–NaCl for heat exchange. The HTF outlet temperature from the storage system should be $\geq 720^{\circ}\text{C}$ to achieve the temperature requirement (700°C) of the s-CO₂ power cycle.

2.3.2. Numerical Simulation Models

To simplify the fabrication of the storage system and reduce the overall LHTES system cost, the entire storage system consists of several LHTES modules, as sketched in Figure 2-1c. Each module contains seven HTF pipes. Standard 2-in. pipes (outer diameter, 60.32 mm; inner diameter, 54.79 mm) were used as the HTF pipes in the storage system. The size of the HTF pipes is based on the capital and operating costs of the LHTES system [4]. Note that the actual piping size and wall thickness also depend on the mechanical properties at the operating conditions, which will be investigated under the parametric study. To ensure most of the PCM undergoes a phase change during the 8-hour charge and 12-hour discharge, each module was about 1.25 m in diameter and 10 m in height, as shown in Figures 2-1a and 2-1b. The distance between each pipe in the module was 0.45 m. The actual diameter of the module will be based on the existing market standard pipe size, which was also considered in the parametric study. To achieve the 2400-MWh storage capacity, about 800 modules are required in the entire storage system. Thus, the diameter of the entire storage system is approximately 35 m, and the height is 10 m based on the height of the storage tank of Gemasolar, a CSP plant in Spain [5]. A single module was used for the current simulations to represent the entire LHTES system because of the adiabatic boundary condition around each module. COMSOL Multiphysics commercial software was used for simulations. The simulation model consisted of 3,527,990 domain elements, 319,688 boundary elements, and 38,260 edge elements. Based on a mesh independence study, these mesh elements were sufficient in the simulations for the LHTES system.

2.3.3. Additional Simulation Parameters

The s-CO₂ flow velocity in the HTF pipes was assumed to be 18 m/s during the charging process, based on the literature data [6]. The CO₂ flow velocity during the discharging process was estimated as 12 m/s (two-thirds of the charging velocity because of the longer discharging time). The inlet CO₂ temperature for the LHTES system during the charging process was set at 880°C . The average outlet CO₂ temperature during the discharging process was about 720°C (to match the s-CO₂ power cycle temperature

requirements). The initial temperature of the storage medium was assumed to be 720°C before the charging process started.

Supercritical CO₂ was used as the HTF; its properties are given in Table 2-1. According to the literature [7], the solar field inlet pressure is about 200 bar for the s-CO₂ power cycle. Thus, the density of the s-CO₂ was estimated on the basis of this pressure [8].

Table 2-1. Thermophysical properties of s-CO₂ [8–10]

	Density (kg/m ³)	Specific heat (J/kgK)	Thermal conductivity (W/mK)	Dynamic viscosity (Ns/m ²)
s-CO ₂	110	1136	0.085 (at 800 °C)	4.2×10^{-5} (under 200 bar at 800°C)

Incoloy 800H was used as the material for the HTF pipes and the prototype container because of its superior oxidation resistance. The thermal properties of the graphite foam–NaCl combination and Incoloy are displayed in Table 2-2. The anisotropic thermal conductivities of graphite foam were considered in the simulations.

Table 2-2. Thermo-physical properties of graphite foam–NaCl and Incoloy 800H [2, 11–13]

	Density (kg/m ³)	Specific heat (J/kg K)	Thermal conductivity (W/m K)	Latent heat of fusion (kJ/kg)
Graphite foam–NaCl	1624	1240 (at melting point of NaCl)	~25 (at 800°C in the high thermal conductivity direction)	414.3
Incoloy 800H	7940	460	24.7 (at 800 °C)	

2.3.4. Validation of Thermal Modeling

As part of the previous LPDP project, extensive thermal simulations were conducted and models were compared with the experimental data. In this section, some of the work is highlighted. To validate this technology and to evaluate its advantages over current technologies, both one-dimensional (1-D) and three-dimensional (3-D) simulation models were developed for an LHTES system, shown in Figure 2-2, based on the commercial software COMSOL. The design of the storage system is a tube-in-shell heat exchanger in which the HTF flows in the pipes and the storage media is in the shell surrounding the HTF pipe.

2.3.4.1. One-Dimensional Thermal Model Verification

Prior to numerical simulations, both 1-D and 3-D COMSOL models were verified. For the 1-D COMSOL model, the results from the numerical simulation using the commercial software COMSOL based on the equivalent heat capacity method were compared with the calculations from a semi-infinite, analytical model [14, 15] for a discharging process. Because of the 1-D nature of the discharge transient for a single HTF pipe, the heat transfer analyses and simulations were conducted only in the radial direction by

neglecting the heat transfer in the angular and axial directions in the storage tank because of the assumption of a uniform HTF temperature along the pipe. A semi-infinite graphite foam–MgCl₂ combination was used for the latent heat storage system. The numerical simulations and the semi-analytical model calculations were conducted for a 219.08-mm HTF pipe. Liquid-state lithium fluoride-sodium fluoride-potassium fluoride (46.5-11.5-42 mol %) (FLiNaK) was considered as the HTF [16]. The heat transfer fluid temperature was set at 500°C for the discharging process. The flow velocity in the HTF pipe was taken as 0.5 m/s. The initial graphite foam–MgCl₂ combination temperature was the MgCl₂ melting temperature of 714°C. The thermo-physical properties of the graphite foam–MgCl₂ combination used in the numerical model in the COMSOL code were the same as those in the analytical model.

The comparison between the 1-D COMSOL numerical simulations and the semi-analytical model calculations is shown in Figure 2-4. In the 1-D COMSOL numerical simulations, a very large radius of 10 m was used for the graphite foam–MgCl₂ combination to denote the semi-infinite domain, and a grid size of 0.66 mm with a node number of 15,000 was used. A finer grid was used to confirm the adequacy of these values. Figure 2-4a shows the interface movement in the graphite foam–MgCl₂ combination. The locations of the solidification front predicted from both methods are close to each other. After the 8-hour discharging process, the solidification front is 0.52 m from the numerical simulations and 0.55 m from the semi-analytical model calculations with a difference of less than 6%. Figure 2-4b shows the temperature profile at various locations in the graphite foam–MgCl₂ combination predicted by the two methods. The temperature decreases first at the location near the HTF pipe ($r = 0.2$ m). After a period of time, the temperature at the location far from the HTF pipe ($r = 0.5$ m) begins to decrease. Since the initial temperature of the graphite foam–MgCl₂ combination is at the melting point of MgCl₂, the temperature of the graphite foam–MgCl₂ combination is maintained at the melting point until the total latent heat is removed. The temperature profiles from both methods agree well.

(a) Location of the solidification front (b) Temperature profiles at various locations

Figure 2-4. Comparison of 1-D COMSOL model and 1-D semi-infinite analytical model

2.3.4.2. Three-Dimensional Thermal Model Verification

For the 3-D COMSOL model, the results of the numerical simulations using the commercial software COMSOL based on the equivalent heat capacity method were compared with the results of the 1-D numerical simulations based on the front-tracking method [17, 18] in which the governing equations combined with boundary conditions were solved by the finite difference method. The comparison was conducted for an 8-hour charging process of a single-HTF-pipe storage system. For the 3-D COMSOL model, the temperature variations in the axial direction (along the HTF pipe), as well as the heat conduction in the radial direction and angular direction, were considered. The 3-D single-pipe model consisted of 518,571 domain elements, 49,416 boundary elements, and 5,928 edge elements, which are sufficient for the current heat transfer simulations. For the 1-D simulation, it was assumed that the temperatures in the HTF and graphite foam–MgCl₂ combination were uniform in both the angular direction and the axial direction, and therefore only thermal gradients in the radial direction were considered. The HTF inlet temperature was maintained at 820°C during the charging process. The initial graphite foam–MgCl₂ combination temperature was 620°C. The radius of the graphite foam–MgCl₂ combination was 0.27 m, and the boundary at a radius of 0.27 m was assumed to be insulated.

The comparison between the 3-D COMSOL numerical simulations and the 1-D front-tracking calculations is shown in Figure 2-5. The temperature profiles in the graphite foam–MgCl₂ combination as well as in the HTF after the 8-hour charging process are shown in Figures 2-5a and 2-5b for the 3-D COMSOL simulation. The HTF temperature decreases along the HTF pipe, as shown in the longitudinal section view in Figure 2-5b. Therefore, the temperatures are higher at the bottom section and relatively lower at the top section in the graphite foam–MgCl₂ combination. The locations of the melting front and the temperature profiles in the graphite foam–MgCl₂ combination at the axial midpoint of the storage tank from the two models are compared in Figures 2-5c and 2-5d, respectively. The melting fronts from both methods agree quite well (within 5%) with each other. The melting front calculated from the 3-D model reaches the outer boundary ($r = 0.27$ m) after 7.35 hours during the charging process, while it takes about 7.3 hours for the melting front to reach the outer boundary according to the 1-D simulation. The temperature profiles at various radii from the 3-D COMSOL model are very close (within 5%) to those from the 1-D simulation model, as shown in Figure 2-5d.

Figure 2-5. Comparison of 3-D COMSOL model and 1-D front-tracking method model

2.3.5. Thermal Performance

Figure 2-6 shows the model calculations for the locations of the melting fronts after an 8-hour charging process or a 12-hour discharging process of LHTES systems without and with graphite foam with NaCl as the PCM. In the phase transition graphs, the red region (1 on the index) indicates complete liquid-state PCM, while the blue region (0) indicates complete solid-state PCM. As shown in Figure 2-6a, the heat transfer performance in the storage system without graphite foam was very poor because of the low thermal conductivity of NaCl (approximately 1 W/mK [2]). Only a very small portion of the PCM around the HTF pipes was melted after the 8-hour charging process because of the large thermal resistance related to heat conduction in NaCl. To ensure that most of the PCM is melted during the charging process (to achieve the 2400-MWh storage capacity), the total number of the HTF pipes in the storage system is about 66,557 for the pure NaCl LHTES

system without the graphite foam, which translates to high capital and operating costs for the TES system.

The heat transfer performance was improved by adding graphite foam in the LHTES system. Because of the high thermal conductivity of graphite foam, the thermal performance in the LHTES system was improved significantly. After the 8-hour charging process (energy storage process), almost all the PCM in the module system was melted, as shown in Figure 2-6b. The melting front in the y-direction moved faster than that in the x-direction because of the anisotropic thermal conductivity of graphite foam. However, because the heat transfer occurred in the circumferential direction through the graphite foam, the front movement and temperature differences between the x- and y-directions were not very significant. Similar front movements occurred in the discharging process (energy retrieval process). After the 12-hour discharging process, all PCM in the module system was solidified, as shown in Figure 2-6c. The high thermal conductivity of graphite foam remarkably increased the heat transfer performance in the LHTES system. This improved heat transfer decreases the total number of HTF pipes needed in the storage system to about 5,607, a reduction factor of 12 compared to the nongraphite foam storage system, and this decrease considerably reduces the costs of the LHTES system.

Figure 2-6. Thermal performance after 8-hour charging or 12-hour discharging

Because of the liquid-like densities of s-CO₂, the heat transfer characteristics of the HTF are excellent, and the heat transfer coefficient of the HTF is very high, with a large Biot number of about 5.4. Thus, the temperature gradient in the radial direction (x- and y-directions) was significant. The PCM close to the HTF pipes melted or solidified first during the charging or discharging process, respectively. Then the heat conducted radially (in x- and y-directions) to melt or solidify the PCM away from the HTF pipes.

The round-trip exergy efficiency (ψ_{round}) was determined by the exergy recovered from the HTF during the discharging process (Ex_{HTF_dis}) as a fraction of the total exergy supplied by the HTF during the charging process (Ex_{HTF_char}), which is expressed as $\psi_{round} = Ex_{HTF_dis} / Ex_{HTF_char}$ [14].

The round-trip exergy efficiencies were calculated for the LHTES systems without and with graphite foam. The low thermal conductivity of NaCl in the pure NaCl system without graphite foam causes the poor heat transfer performance in the storage system. Only a very small portion of PCM is melted after the charging process, which results in the low latent heat storage in the system. Therefore, the exergy efficiency of the NaCl LHTES system is quite low, just 48.4%. The high thermal conductivity of graphite foam significantly increases the heat transfer performance in the graphite foam–NaCl LHTES system. Thus, the exergy efficiency of the graphite foam–NaCl LHTES system is increased to 96.3%. Furthermore, the average HTF outlet temperature from the LHTES system during the discharging process is about 720°C, which meets the temperature requirements (650–700°C) of the s-CO₂ power cycle. Therefore, the graphite foam–PCM LHTES system is capable of large-scale energy storage in the CSP plant.

Table 2-3. Exergy efficiency for LHTES without and with graphite foam

LHTES system	Round-trip exergy efficiency, ψ_{round}
<i>Without graphite foam</i> Storage medium: NaCl 8-hour charging, inlet temperature 880°C Average outlet temperature 878.3°C $V_{HTF_char} = 18$ m/s 12-hour discharging, inlet temperature 720°C Average outlet temperature 720.8°C $V_{HTF_dis} = 12$ m/s	48.4%
<i>With graphite foam</i> Storage medium: graphite foam–NaCl 8-hour charging, inlet temperature 880°C Average outlet temperature 870.1°C $V_{HTF_char} = 18$ m/s 12-hour discharging, inlet temperature 715°C Average outlet temperature 725.1°C $V_{HTF_dis} = 12$ m/s	96.3%

2.3.6. Analysis Using Graphite Foam–MgCl₂

This analysis was also conducted for MgCl₂ PCM. Graphite foam–MgCl₂ composite was used as the storage medium. Supercritical CO₂ was considered as the HTF through the pipes. Each modular container was 18 in. in outer diameter. The simulation model consisted of 1,955,793 domain elements, 294,244 boundary elements, and 39,414 edge elements. Based on mesh independence studies, these mesh elements were sufficient in

the simulations for the LHTES system. The simulation model mesh structure is displayed in Figure 2-7.

Figure 2-7. Mesh structure in the simulation model

The anisotropic thermal conductivity of the composite was considered in the simulations. The graphite foam acts as a thermal conductivity enhancement additive to significantly improve the effective thermal conductivity of the graphite foam–PCM composite. Figure 2-8 displays the thermal conductivity values of two types of graphite foams and the PCM-infiltrated foams measured through the Flash Diffusivity Analyzer (FDA). The graphite foam can increase the effective thermal conductivity of the composite by >10–20 times at the elevated temperature (i.e., operating temperature of the LHTES system) compared to that of pure MgCl₂ (i.e., thermal conductivity of ~0.3 W/mK). Moreover, the thermal conductivity of graphite foam–MgCl₂ in the z-direction (axial direction) was about two times higher than that in the x-y plane, as shown in Figure 2-8.

Figure 2-8. Thermal conductivity of (a) high-density (HD) graphite foam (open porosity of 55%) and (b) low-density (LD) graphite foam (open porosity of 90%) with and without MgCl₂ in the z-direction and x-y plane measured by FDA [19]

The latent heat of fusion of the graphite foam–PCM composite was measured by differential scanning calorimetry (DSC). Figure 2-9 shows the measured latent heat

values of MgCl₂-infiltrated HD and LD foams. It was found that the presence of graphite foam did not affect the melting and freezing temperature of MgCl₂. The latent heat of fusion of the graphite foam–MgCl₂ composite was in the range of 225.2–379.6 kJ/kg based on different types of graphite foam. Moreover, the heating–cooling thermal cycles were repeated for 100 cycles. The DSC results were repeatable [19].

Figure 2-9. Latent heat of fusion value of (a) HD graphite foam–PCM composite and (b) LD graphite foam–PCM composite through DSC measurements [19]

Supercritical CO₂ was used as the HTF; its properties are given in Table 2-1. According to the literature [3], the solar field inlet pressure is about 200 bar for the s-CO₂ power cycle. Thus, the density of the s-CO₂ was estimated on the basis of this pressure [9]. The HTF flows from the top of the modular container to the bottom during the charging process, and from the bottom to the top during the discharging process. Note that for a CSP plant, HTF from the receiver could also be a high-temperature salt.

Tables 2-4 and 2-5 illustrate the charging and discharging conditions, the geometries, and the performances of the LHTES system in the COMSOL simulations. Approximately 4,383 modular containers with about 30,681 HTF pipes are required to achieve a 200-MW_{th} storage capacity of the LHTES system. The round-trip exergy efficiency of the graphite foam–MgCl₂ LHTES system was very high, more than 92.5%, based on the simulations because of the high thermal conductivity of the graphite foam component.

Table 2-4. Charging and discharging conditions of the LHTES system with various types of HTFs

Condition	s-CO ₂ at 200 bar	
	Charging Process	Discharging Process
Flow velocity, m/s	1.5	0.2–0.3
HTF inlet temperature, °C	750	520
HTF outlet temperature, °C	704.2	≥700

Table 2-5. Geometry and exergy efficiency of the LHTES system with various types of HTFs

	s-CO ₂ at 200 bar
Modular container ^a outer diameter, in.	18
Graphite foam open porosity	90%
Number of modules	4383
Number of HTF pipes	30681
Energetic efficiency of the LHTES system	99.5%
Round-trip exergy efficiency of the LHTES system	92.5%

^a Modular container wall thickness: 0.375 in.

The locations of the melting or solidification fronts in the modules during the charging or discharging processes are displayed in Figure 2-10. At the same time point, larger amounts of PCM melted during the charging process when graphite foam was applied in the system compared to the system without graphite foam. Thus, more HTF pipes would be required in the pure PCM system for heat exchange to fully utilize the storage medium.

Figure 2-10. Locations of the melting/solidification fronts in the modules: (a) melting fronts at 4 hours in the charging process in the pure PCM system; (b) melting fronts at 4 hours in the charging process in the graphite foam–PCM composite-based LHTES system; and (c) solidification fronts at 4 hours in the discharging process in the graphite foam–PCM LHTES system

Figures 2-11 and 2-12 show the temperature distribution in the module during the charging and discharging processes, respectively. For the charging process, the temperature of the graphite foam–MgCl₂ composite at the top of the module increased first, and then the heat spread down to the bottom because the HTF flowed from the top to the bottom. For the discharging process, the flow direction reversed. Therefore, the

storage medium at the bottom of the module solidified first. It was determined that the fluid flow direction made the molten PCM stay on the top of the module instead of at the bottom to avoid the rapid increase of pressure inside the container because of the volume expansion of molten PCM during the phase change process.

Figure 2-11. Temperature distribution in the modular container at various times and elevations during the charging process: (a) cross-section view of temperature at the middle of the module ($z = 5$ m) at 4 hours; (b) overall view of the module temperature distribution at 4 hours; (c) cross-section view of temperature at the bottom of the module ($z = 0$ m) at 8 hours; and (d) overall view of the module temperature distribution at 8 hours

Figure 2-12. Temperature distribution in the modular container at various times and elevations during the discharging process: (a) cross-section view of temperature at the middle of the module ($z = 5$ m) at 4 hours; (b) overall view of the module temperature distribution at 4 hours; (c) cross-section view of temperature at the top of the module ($z = 10$ m) at 8.3 hours; and (d) overall view of the module temperature distribution at 8.3 hours

Based on the investigations of the full-scale system, the graphite foam could significantly improve the heat transfer performance of the LHTES system. The graphite foam can help accelerate the melting/solidification process for efficient thermal energy storage/release, as shown in Figure 2-10. Hence, use of graphite foam results in an eight-fold reduction in the number of required HTF tubes compared to a PCM-only system, therefore leading to significant cost reductions in the CSP plant. Furthermore, the graphite foam–PCM composite could achieve a system energetic efficiency of more than 99% and a round-trip exergy efficiency of more than 90%–95% to meet the U.S. Department of Energy (DOE) target for the LHTES system thermal performance, which cannot be accomplished by using pure PCM. Finally, the analyses using various HTFs such as sodium metal, chloride salt, and helium gas are presented in Appendix A.

2.3.7. Parametric Studies

In order to design and optimize the full-scale LHTES system in the CSP plant, various system parameters were investigated, including (1) the open porosity of the graphite foam; (2) the material, diameter, and wall thickness of the HTF pipe; (3) the thickness of the interface gap if there is no brazing between HTF pipe and graphite foam; and (4) the thickness of the container wall; to study their effects on the energetic efficiency and the exergy efficiencies of the LHTES system.

Numerical simulations were conducted for the modular LHTES system to study the system parameter effects. The time of the charging process (energy storage process) was 8 hours, while the time of the discharging process (energy retrieval process) was 12 hours for the full-scale LHTES system in the CSP plant. The base case of the simulation model was 84% open-porosity graphite foam (from Koppers Inc.) in the storage system with 2-in. Incoloy 800H pipe (pipe wall thickness of 0.436 in., double extra heavy) as HTF pipes and 48-in. stainless steel pipe (pipe wall thickness of 0.375 in., standard wall thickness for 48-in. pipe) as modular containers to hold the storage medium. The parameters were varied based on the base case of the model to study their effects on the energetic and exergy efficiencies of the system.

2.3.7.1. Graphite Foam Open-Porosity Effect

Graphite foams with various open porosities in the range of 55%–95% are currently available in the market. Simulations were conducted for 55%, 84%, and 90% open-porosity graphite foams from POCO Inc., Koppers Inc., and GrafTech, respectively. The higher open porosity leads to lower thermal conductivity and lower density of graphite foam. Nevertheless, the higher open-porosity graphite foam can be infiltrated by larger amounts of PCM, which enhances the latent heat of fusion for a certain volume of graphite foam–PCM combination. However, the high open porosity of graphite foam causes a decrease in the overall thermal conductivity of the graphite foam–PCM combination, which results in relatively lower heat transfer performance in the storage system. Table 2-6 summarizes the overall thermal conductivity and the latent heat of fusion of the graphite foam–NaCl combination with various graphite foam open porosities.

Table 2-6. Thermal conductivities and latent heat of fusion of graphite foam–NaCl combination

Graphite foam open porosity (%)	Thermal conductivity (W/mK) (at 700°C in the high-thermal-conductivity direction)	Latent heat of fusion [2] (kJ/kg)
55	81	219.5
84	24	376.9
90	22.5	414.4

Figure 2-13 shows the energetic and exergy efficiencies of various open-porosity graphite foam. Higher open porosity slightly improves the efficiencies of the LHTES system because of the higher latent heat density in the system. According to the simulation results, displayed in Figure 2-13, the energetic and exergy efficiencies of the LHTES system with 84% open-porosity graphite foam can reach 100% and 95%, respectively. Therefore, the graphite foam with an open porosity of more than 84% would be chosen for the storage system to reach the performance targets.

Figure 2-13. Efficiency of full-scale LHTES system as a function of the open porosity of graphite foam

2.3.7.2. Effects of HTF Pipe Material, Diameter, and Wall Thickness

Two different HTF pipe materials, stainless steel and Incoloy 800H, were studied for their effects on storage system efficiencies. Figure 2-14 shows that the energetic and exergy efficiencies do not change with different HTF pipe materials because Incoloy 800H and stainless steel have similar thermal properties. Both HTF pipe materials can help reach 100% energetic efficiency and 95% exergy efficiency. Therefore, both materials can be considered as potential candidates for HTF pipe material based on their heat transfer properties. Nevertheless, with respect to durability, Incoloy 800H shows better performance at elevated temperatures.

Figure 2-14. Efficiency of full-scale LHTES system with different HTF pipe materials

Furthermore, various HTF pipe diameter sizes were also studied for their effects on system efficiencies. Two-in., 3-in., and 4-in. standard pipe sizes (with double extra heavy wall thickness) were investigated. Figure 2-15 displays the energetic and exergy efficiencies of the LHTES system with the three pipe sizes. The pipe size did not affect the energetic and exergy efficiencies significantly. Based on the simulation results, larger HTF pipe size could slightly increase exergy efficiency. All three HTF pipe sizes could meet the exergy efficiency target of more than 95%. Nevertheless, larger pipe size would increase the capital cost and the pumping power of the system.

Figure 2-15. Efficiency of full-scale LHTES system with various HTF pipe sizes

Moreover, the HTF pipe wall thickness was also considered as one of the effects on system efficiency. Figure 2-16 shows the energetic and exergy efficiencies with various HTF pipe wall thicknesses (2-in. pipe was considered in the analysis). Larger HTF pipe wall thickness would slightly decrease the system exergy efficiency because of the larger

thermal resistance of the HTF pipe wall, but it still meets the exergy efficiency target of more than 95% even for the largest wall thickness (double extra heavy, 0.436 in.).

Figure 2-16. Efficiency of full-scale LHTES system with various HTF pipe wall thicknesses

Because of the high pressure of s-CO₂ in the HTF pipe (about 200 bar), the pipe wall thickness should be at least Schedule 160 (0.343 in.) for the 2-in. pipe to withstand such high pressures of s-CO₂ HTF [20].

2.3.7.3. HTF Pipe and Graphite Foam Interface Gap Thickness Effect

The gap thickness of the interface between the HTF pipe and the graphite foam is another important parameter affecting the heat transfer performance in the LHTES system. When the brazing process is conducted between the HTF pipe and the graphite foam, there is no gap at the interface. If the brazing process is not conducted, the gap between the HTF pipe and the graphite foam is filled with NaCl, which forms a thermal resistance layer because of the low thermal conductivity of NaCl material (about 1 W/mK [2]). This thermal resistance layer affects the heat transfer performance of the LHTES system, reducing the heat transfer rate in the system. The PCM (NaCl) in the module is not completely melted during the 8-hour charging process, and this decreases the energetic and exergy efficiencies of the system.

Figure 2-17 illustrates the interface gap effect on the system energetic and exergy efficiencies. Without the gap (0 mm thickness), the energetic and exergy efficiencies reach 100% and 95%, respectively. With the increase in the gap thickness (0.5 mm and 1.0 mm), the system efficiencies decrease, especially for larger gap thickness (1.0 mm), as shown in Figure 2-11.

Figure 2-17. Efficiency of full-scale LHTES system as a function of interface gap thickness

2.3.7.4. Modular Container Wall Thickness Effect

Standard 48-in. pipe was used as the modular container to hold the storage medium, the graphite foam–NaCl combination. Stainless steel was applied as the modular container material. A standard wall thickness (0.375 in.) and an extra heavy wall thickness (0.5 in.) for 48-in. pipe were applied in the simulation models. Figure 2-18 illustrates the energetic and exergy efficiencies of the LHTES system with different container wall thicknesses. Larger container wall thicknesses decrease the energetic and exergy efficiencies of the storage system because of the reduction of the storage medium in the modular container. Therefore, it is not necessary to use pipe with a larger wall thickness as the modular container. The standard wall thickness (0.375 in.) for the 48-in. pipe is adequate for reducing the cost of the material.

Figure 2-18. Efficiency of full-scale LHTES system with various container wall thicknesses

In summary, various system parameters affect the energetic and exergy efficiencies of the LHTES system: the open porosity of the graphite foam, the size and diameter of the HTF pipe, the thickness of the interface gap between the HTF pipe and the graphite foam, and the wall thickness of the modular container. According to the parametric studies, (1) the open porosity of the graphite foam should be >84%; (2) the optimum size of the HTF pipe should be 2 in. with a schedule 160 wall thickness considering the combined effects of exergy efficiency, high operating pressure (200 bar) of s-CO₂, and costs; (3) the thickness of the interface gap between the HTF pipe and the graphite foam should be less than 0.5 mm based on the system heat transfer performance; and (4) the optimum size of the modular container is 48-in. pipe (~1.2 m outer diameter) with a standard wall thickness (0.375 in.). Stainless steel should be the container material to reduce the capital cost.

2.3.8. Comparison with Current LHTES Systems

Most of the current PCM-based storage technologies are tube-in-shell configurations. The key performance restriction is the low thermal conductivity of the PCMs. To improve the thermal conductivity of the PCMs, the most common approach is the finned system. Here the HTF pipe has fins extended into the PCM. These fins are metallic and provide enhanced heat transfer. An experimental demonstration was recently conducted by Miliozzi et al. [21] using this approach.

To establish the performance ranges for tube-in-shell type LHTES systems, three different LHTES configurations were considered to compare their heat transfer performance through the COMSOL heat transfer simulations as well as the energetic and exergy efficiencies of the systems. The first type of LHTES system has only the PCM (NaCl) in the system without graphite foam. The second type of LHTES system has fin structures on the HTF pipe to improve the thermal performance in the PCM (no graphite foam in the system). The third type of LHTES system has graphite foam embedded in the PCM to enhance the effective thermal conductivity of the graphite foam–NaCl combination in order to improve the thermal performance of the LHTES system (without fin structures on the HTF pipe). Note that the choice of materials (fin material and so on) was for comparison purposes.

The fin structures for the second type of LHTES system were built based on the annular fin model from Miliozzi et al. [20]. Figure 2-19 shows the annular fin geometry around the HTF pipe in the LHTES system for the heat transfer simulations. In the simulation model, the fin thickness is 20 mm; the fin length (from the base to the tip of the fin) is about 30 mm; and the distance between each fin is 40 mm. The fin structure in this model is based on the model of Miliozzi et al. [20] and scaled up for the 10-m-high seven-pipe modular LHTES system. To save computational times, the basic LHTES model in the simulations is about 16 in. in diameter and 1-m in height with one HTF pipe at the center (unlike the seven-pipe modular system in the full-scale modular LHTES). Supercritical-CO₂ was considered as the working fluid inside the HTF pipe for heat exchange in the simulations. The center HTF pipe is a standard 2-in. pipe with a wall thickness of 0.436 in. for the s-

CO₂ application. The s-CO₂ flow velocity was 12–18 m/s in the pipe, the same as that in the seven-pipe modular system for the full-scale LHTES system [22]. Table 2-7 lists the charging/discharging conditions.

Figure 2-19. Fin structures modeled around the HTF pipe in the LHTES system

Table 2-7. Charging and discharging conditions for the LHTES systems

	s-CO ₂ HTF inlet temperature (°C)	s-CO ₂ HTF flow velocity (m/s)	LHTES initial temperature (°C)	Time (hour)
Charging process	880	18	720	8
Discharging process	720	12	880	12

Figures 2-20 and 2-21 display the heat transfer performance in the three types of LHTES configurations. The results are at the mid-section of the LHTES systems after 4 hours of charging/discharging. The system with PCM only has very poor thermal performance because of the low thermal conductivity of NaCl (only ~ 1 W/mK). When the fin structures are included around the HTF pipe, the thermal performance in the PCM is improved because of the increased heat transfer surfaces. Nevertheless, the improvement is not significant compared to that of the graphite foam-PCM system, as shown in Figures 2-20c and 2-21c. The graphite foam-PCM combination enhances the heat transfer performance significantly in the LHTES system. *The heat transfer rate in the graphite foam-PCM system is approximately three times higher than that in the PCM-plus-fin system, which makes the graphite foam-PCM system the best candidate for the LHTES system in the CSP plant application.*

Figure 2-20. Heat transfer performance in three LHTES configurations (cross-section view at the middle of the LHTES system)

Figure 2-21. Heat transfer performance in three LHTES configurations during the discharging process (cross-section view at the middle of the LHTES system)

Table 2-8 illustrates the energetic and exergy efficiencies of these three types of the LHTES systems. Case 1 is the NaCl system without the graphite foam and the fin structures around the HTF pipes. Because of the very low thermal conductivity of NaCl, most of the NaCl in the system was not melted during the 8-hour charging process. It results in the low energetic and exergy efficiencies of the system, as displayed in Table 2-6. Case 2 is the NaCl system (no graphite foam) with the fin structures around the HTF pipe. The fin structures could help increase the heat transfer performance in the NaCl system. Therefore, the energetic and exergy efficiencies are improved compared to case 1 (no fin structures). Nevertheless, it does not reach the targets for energetic efficiency, >99%, and exergy efficiency, >95%, yet. Case 3 is the graphite foam–NaCl system (no fin structures). The graphite foam can significantly enhance the effective thermal conductivity of the graphite foam–PCM combination and therefore improve the heat transfer performance in the LHTES system remarkably, as displayed in Figures 2-20 and 2-21. The NaCl in the system was completely melted during the 8-hour charging process, which increases its energetic efficiency to approximately 100% and exergy efficiency to 96.7%. Thus, the graphite foam–PCM LHTES system is the most promising storage system for the CSP plant to reduce the levelized cost of energy (LCOE).

Table 2-8. Energetic and exergy efficiencies of the three LHTES configurations

	Energetic efficiency	Exergy efficiency
Case 1: NaCl LHTES system (no graphite foam, no fin structures) 8-hour charging, inlet temperature 880°C average outlet temperature 879.7°C $V_{HTF_char} = 18$ m/s 12-hour discharging, inlet temperature 720°C average outlet temperature 720.2°C $V_{HTF_dis} = 12$ m/s	66.7%	64.5%
Case 2: NaCl LHTES system (no graphite foam), with fin structures 8-hour charging, inlet temperature 880°C average outlet temperature. 879.5°C $V_{HTF_char} = 18$ m/s 12-hour discharging, inlet temperature 720°C average outlet temperature 720.4°C $V_{HTF_dis} = 12$ m/s	80.0%	77.3%
Case 3: Graphite foam–NaCl, no fin structures 8-hour charging, inlet temperature 880°C average outlet temperature 878.6°C $V_{HTF_char} = 18$ m/s 12-hour discharging, inlet temperature 720°C average outlet temperature 721.4°C $V_{HTF_dis} = 12$ m/s	100.0%	96.7%

This analysis demonstrates the significant performance advantages of the graphite foam–PCM LHTES system over the current LHTES system employed for demonstration.

2.3.9. Heat Loss and Energetic/Exergy Efficiency

For a full-scale storage system, it is key in the energetic and exergetic analysis to take the heat loss into consideration. Based on insulation properties, temperature of storage system operation, and configuration of the LHTES system, heat losses were estimated. Subsequently, this heat loss was considered in the energy analysis.

2.3.9.1. Heat Loss Calculation

Current two-tank SHTES system

$T_{ambient\ air} = 40^{\circ}\text{C}$; tank size, 37.2 m (diameter) $\times$ 14 m (height) [23]

Hot tank, 390°C ; cold tank, 290°C [2, 21]

Power cycle, 15 MW_e [2]; thermal-to-electric conversion efficiency, 37.5% [23, 24]

TES system, 40 MW_t

1% heat loss (Q_{loss}), 0.4MW_t

Heat loss energy balance (two tank)

$$Q_{loss} = 2 \times hA(T_s - T_{ambient\ air}) = kA \frac{T_{hot_tank} - T_s}{\Delta L_{hot_tank}} + kA \frac{T_{cold_tank} - T_s}{\Delta L_{cold_tank}}$$

where

$$\Delta L_{hot_tank} = 373\text{ mm}$$

$$\Delta L_{cold_tank} = 300\text{ mm} [25]$$

$$k = 0.085\text{ W/mK (calcium silicate block insulation) [24, 26]}$$

T_s = surface temperature.

$$T_s = 47.2^{\circ}\text{C}, h = 10.2\text{ W/m}^2\text{ K}$$

One-tank LHTES system

$T_{ambient\ air} = 40^{\circ}\text{C}$; tank size, 35 m (diameter) $\times$ 10 m (height)

Power cycle, 100 MW_e; thermal-to-electric conversion efficiency, 50%

TES system, 200 MW_t

1% heat loss (Q_{loss}), 2.0 MW_t

Heat loss energy balance (single tank)

$$Q_{loss} = hA(T_s - T_{ambient\ air}) = kA \frac{T_{LHTES} - T_s}{\Delta L}$$

where

$$k = 0.12\text{ W/mK [26]}$$

$$T_s = 135^{\circ}\text{C}, \Delta L = 82\text{ mm}$$

Note: If $\Delta L = 670\text{ mm}$ (calculated based on the insulation thickness for the current SHTES)

$$T_s = 53.1^{\circ}\text{C}, Q_{loss} = 0.3\text{ MW (0.15\% heat loss)}$$

(Note: The 1% heat loss from the TES system was obtained from the literature [27].)

2.3.9.1. Energetic and Exergy Efficiency

Using the heat losses estimated above for the single LHTES system, an energy analysis was carried out for the LHTES system for both single-tank and seven-pipe module designs. The results are presented below.

Single-tank system

Heat loss around the seven-pipe hexagon storage geometry, 1%

s-CO₂ HTF temperature during charging, inlet 880°C and outlet 861.4°C

s-CO₂ temperature during discharging, inlet 715°C and outlet 733.4°C

HTF velocity during charging, 18 m/s; HTF velocity during discharging, 12 m/s

Energetic efficiency, 99.1%; exergy efficiency, 95.8%

(a) Phase transition between solid-state and liquid-state PCM (b) Temperature distribution in the storage system

Figure 2-22. Single-tank system

Seven-pipe modular system

Heat loss around the seven-pipe module system, 1%

s-CO₂ HTF temperature during charging, inlet 880°C and outlet 861.5°C

s-CO₂ temperature during discharging, inlet 715°C and outlet 733.5°C

HTF velocity during charging, 18 m/s; HTF velocity during discharging, 12 m/s

Energetic efficiency, 99.8%; exergy efficiency, 96.5%

(a) Phase transition between solid-state and liquid-state PCM (b) Temperature distribution in the storage system

Figure 2-23. Seven-pipe modular system

These results show that, after incorporating the heat loss, the energetic and exergy efficiencies are greater than 99% and 95%, respectively.

3. Experimental Investigation of Effects of HTF Pipe Gaps on LHTES Systems

As discussed previously, in the LPDP project, the HTF pipe was brazed to the graphite foam. However, brazing adds not only another process step but also cost to the overall LHTES system. So this task focused on establishing the thermal performance of the LHTES module without any HTF pipe-to-graphite brazing and comparing it with the simulations. In addition, there were couple of fabrication issues from the LPDP project that were first resolved.

3.1. Critical Issues of Laboratory-Scale Prototypes

Issues related to the fabrication of laboratory-scale prototypes were identified from the previous SunShot project. Two of them have critical importance to the fabrication of future laboratory-scale prototypes and are discussed in the following paragraphs.

3.1.1. Thermocouple Feed-Through Body Sealing

In the initial design and fabrication of laboratory-scale prototypes, sealing between the thermocouple feed-through bodies and the top cap was achieved using high-temperature DEACON 8875. It was found that while the sealing material worked generally well, there was a tendency for it to develop small leakages. Because of these leakages, the system vacuum could not be maintained. A leak-sealing method was developed, and leak-sealing welding was conducted to fix the problem. Figure 3-1 shows one of the feed-through bodies being leak-sealing welded by using an adaptor. Two other feed-through bodies were welded in the same way.

Figure 3-1. Sealing issue between thermocouple feed-through bodies and top cap

3.1.2. Leakage through HTF Pipes

In the initial design and fabrication of laboratory-scale prototypes, the HTF pipe was a commercial Incoloy 800H tubing with an outside diameter of 1/2 in. and a wall thickness of 0.028 in. The very thin wall thickness of the tubing caused welding difficulty because

of uneven heating. The thin-wall tubing was found to have a tendency to completely melt through its wall during welding because of overheating. Figure 3-2 shows a ring around the inner surface of the HTF pipe at the welding section indicating overheating. Several prototypes had problems caused by this issue: (a) one prototype was found to have a leakage of the HTF pipe during the welding process, (b) one prototype was found to have a leakage of the HTF pipe during the vacuum checking process, and (c) one prototype was found to have a blockage of the HTF pipe during charging experiments caused by the melting salt leaking into the HTF pipe. A method was developed to fix the problem: welding an inner tubing at both ends to the HTF tubing.

Figure 3-2. HTF pipe leakage caused by welding

3.2. Overall Design of Prototypes

The detailed design of laboratory-scale prototypes was revised based on the targets of the current project and the experience gained from the previous project. Figure 3-3 shows schematically some design considerations including the following:

- Type 304 stainless steel (SS 304) was used as the material for the top cap, the bottom cap, and the container of the prototypes.
- A nickel alloy was used as the material for the HTF pipe. The dimensions of the HTF pipe were 5/8 in. in diameter and 0.049 in. in wall thickness. The thick wall was chosen to eliminate the leaking caused by welding the HTF tubing to the top cap and the bottom cap to mitigate tube deformation caused by tightening of Swagelok adapters.
- Thermocouple feed-through bodies were fixed to the top cap with 1/8-in. National Pipe Tapered (NPT) thread connectors. In addition, instead of high-temperature

bonding, welding was used between the feed-through bodies and the top cap to make them leak-tight.

- No brazing between the HTF pipe and the graphite foam was done.
- The most important modification was the elimination of welding between the HTF pipe and the top and bottom caps. As shown in Figure 3-3, Swagelok connectors were welded to the top cap and the bottom cap, and the HTF pipe was locked in position by tightening the connectors. The revised design completely eliminates the requirements of HTF pipe welding, which is usually difficult because of its thin wall thicknesses.

Figure 3-3. Design of “inexpensive” SS 304 prototype

3.3. Fabrication of Prototypes

3.3.1. Synthesis and Characterization of NaCl–KCl Eutectic Salt

Since there were safety issues with the emanation of chloride fumes during graphite infiltration with MgCl₂ during the LPDP project, the infiltration vendor did not agree to make the runs. Thus there was a delay in the project timeline, and a new vendor was identified. However, this vendor had limitations in terms of the temperature that could be achieved in the infiltration system, so a lower temperature PCM (NaCl–potassium chloride [KCl]) eutectic was used instead of MgCl₂. The NaCl–KCl eutectic salt melts at 657°C and has a heat of fusion of ~230 J/g. Since the NaCl–KCl eutectic salt is not commercially available, we synthesized the salt in-house using the following procedure:

- Heat NaCl and KCl powders at 125°C in a vacuum oven at ~0.1 MPa for 60 minutes.

- (b) Mix the two salts in appropriate proportions of 0.506 mole NaCl and 0.494 mole KCl in a mortar and pestle for 30 minutes.
- (c) Further mix the NaCl–KCl mixture on a roller mixer for 60 minutes.
- (d) Dry the NaCl–KCl mixture in the vacuum furnace for 60 minutes.
- (e) Keep the dried NaCl–KCl mixture in a sealed container.

In-house synthesized powder was characterized for heat of fusion and melting point using a simultaneous DSC-TGA system. As shown in Figure 3-4, the measured melting point was 655.36°C, and the measured heat of fusion was 231.4 J/g. These values are fairly close to the literature-reported values. More than 10 kg of the eutectic salt was synthesized and sent to the infiltration vendor.

Figure 3-4. DSC curve for in-house synthesized NaCl–KCl PCM salt

3.3.2. Machining of Prototype Parts

All parts of the prototype, including container cylinder, top plate, bottom plate, center tube, graphite foam, and associated adaptors, were designed to ensure a good fit between the graphite foam–PCM and the container cylinder/the center tube. Machining of the prototype parts was completed according to the design criteria. Figure 3-5 shows the machined prototype parts.

Figure 3-5. Machined prototype parts

After the bottom plate was welded to the container cylinder, the graphite foam was brazed to the bottom plate. The purpose of the brazing is to prevent any movement between the graphite foam and the container cylinder during infiltration. There is no brazing between the graphite foam and the container cylinder/the center tube. Figure 3-6 shows one of the “inexpensive” stainless-steel prototypes that was machined/welded/brazed and ready for infiltration.

Figure 3-6. Machined and welded prototype ready for infiltration

3.3.3. Infiltration of Graphite Foam with NaCl–KCl Eutectic Salt

3.3.3.1. Infiltration

The infiltration vendor successfully completed infiltration for one of the prototypes. Figure 3-7 shows the prototype after infiltration. The prototype was carefully weighed before and after infiltration to determine the infiltration rate. Based on preliminary weight measurements, the estimated infiltration rate was >80% (based on the solid density of the PCM). This run demonstrated that other PCMs can be successfully infiltrated into the graphite foam.

Figure 3-7. Infiltrated prototype with NaCl–KCl eutectic PCM

3.3.3.2. Cleaning, Instrumentation, and Sealing of the Prototype

To prepare the prototype for the next step of thermocouple instrumentation and experimental testing, the infiltrated prototype was completely cleaned: the outside of the

bottom plate, container cylinder, and HTF pipe; the inside of the HTF pipe; and the top space of the graphite foam–PCM composite. Figure 3-8b shows the cleaned prototype.

(a) After infiltration and before cleaning

(b) After infiltration and cleaning

Figure 3-8. Cleaned prototype with NaCl–KCl eutectic PCM

3.3.3.3. Thermocouple Installations

Temperature-monitoring thermocouples were then installed in the storage medium of the prototype. The final step for the fabrication of the prototype was the seal-welding of the top plate to the container cylinder. Vacuum testing was conducted after the welding to verify completely sealing.

3.4. Experimental Testing

3.4.1. Prototype Installation into the Test Loop

The prototype installation into the test loop included the following main steps:

- (a) Attaching pipes with appropriate lengths to the top and the bottom of the prototype center HTF pipe;
- (b) Attaching a pipe with an appropriate length to the prototype vacuum port;
- (c) Insulating the prototype;
- (d) Loading the prototype into the test furnace;
- (e) Connecting the prototype to the test loop;
- (f) Connecting the prototype vacuum port to the vacuum pump line; and
- (g) Pumping down the prototype vacuum for leak check.

Figure 3-9 shows the prototype installation in the test furnace.

Figure 3-9. Installation of prototype in test furnace

3.4.2. Moisture and Oxygen Removal

After the prototype was installed in the furnace, the moisture and oxygen removal procedure was conducted according to the schematic shown in Figure 3-10 through the following steps.

- (a) Pump down the prototype vacuum and monitor the vacuum level change during a 12-hour period to check any potential leakage.
- (b) Heat the prototype to 275°C; keep the prototype at 275°C for 4 hours; and pump down the prototype vacuum periodically to remove moisture.
- (c) Heat the prototype to 575°C; keep the prototype at 575°C for 24 hours; and pump down the prototype vacuum periodically to remove moisture.

After the moisture removal procedure, the prototype was pressurized with argon to approximately 10 psig and then vacuumed down to approximately 500 mTorr five times to create an argon environment in the prototype.

Figure 3-10. Furnace setting for moisture and oxygen removal from prototype

3.4.3. Charging and Discharging

Charging and discharging were generally conducted in one cycle, as shown schematically in Figure 3-11, through the following steps.

- Preheat the furnace to 612°C.
- Switch the heating tapes for heat transfer pipes; turn on the air pump; and set up the heater controller to give an air inlet temperature of the prototype of approximately 645°C.
- Wait for approximately 2 hours for the prototype to reach an equilibrium state.
- Start the charging procedure by increasing the heater controller setting to give an air inlet temperature of the prototype of approximately 743°C.
- After the charging procedure, increase the furnace temperature to 672°C and reduce the heater controller setting to give an air inlet temperature of the prototype of approximately 700°C.
- Wait for approximately 1 hour for the prototype to reach an equilibrium state.
- Start the discharging procedure by decreasing the the heater controller setting to give an air inlet temperature of the prototype of approximately 542°C.

Figure 3-11. Furnace and heat transfer air settings during charging/discharging tests

3.5. Experimental Results

Three charging and discharging tests were conducted on the prototype. The detailed furnace temperature settings and heat transfer air temperatures are listed in Table 3-1. During each charging or discharging process, the air flow rate was not adjusted; however, some fluctuations existed because of air temperature changes. All readings from various sensors including the thermocouples, the air flow meter, and the air pressure transducer were automatically logged into the data acquisition computer in a predetermined time interval of approximately 6s.

Table 3-1. Temperature settings for charging and discharging tests

Test	Charging		Discharging	
	Furnace temperature	Heat transfer air temperature	Furnace temperature	Heat transfer air temperature
1	612°C	755°C	672°C	415°C
2	612°C	743°C	672°C	542°C
3	612°C	743°C	672°C	542°C

The temperature changes based on measurements from thermocouple 4 during charging and discharging tests are plotted in Figures 3-12 and 3-13, respectively. In the charging process, the KCl–NaCl mixture changed from the solid state to the liquid state through three thermal energy storage stages. First, the solid KCl–NaCl–graphite foam was heated and its temperature increased as a result of sensible heat energy storage. Second, when the temperature of the KCl–NaCl mixture was high enough, the solid KCl–NaCl mixture started melting and latent heat energy storage occurred. Third, when the melting process completed, the liquid KCl–NaCl mixture and graphite foam was heated and its temperature increased again as a result of sensible heat energy storage.

In the discharging process, the KCl–NaCl mixture changed from the liquid state to the solid state through three thermal energy release stages. First, the liquid KCl–NaCl–graphite foam was cooled and its temperature decreased as a result of sensible heat energy release. Second, when the temperature of the KCl–NaCl mixture was low enough, the liquid KCl–NaCl mixture started solidification and latent heat energy release occurred. Third, when the solidification process completed, the solid KCl–NaCl–graphite foam was cooled and its temperature decreased again as a result of sensible heat energy release. Figures 3-12 and 3-13 also show that the charging and discharging curves for tests 2 and 3 with similar furnace and heat transfer air temperatures are very similar, showing stable thermal performance of the prototype.

Figure 3-12. Temperature as a function of time for charging tests

Figure 3-13. Temperature as a function of time for discharging tests

3.6. Comparisons with Numerical Simulations

For each charging/discharging test of the prototype, numerical simulations were conducted according to the test conditions, and the experimental data and the simulation results were compared. Figures 3-14 and 3-15 display the comparisons of the temperature profiles for charging and discharging, respectively. Figures 3-14 and 3-15 show that the simulation results match the experimental data quite well. For the charging process shown in Figure 3-14, the temperature differences between the simulation results and the experimental data are generally within 5°C. For the discharging process shown in Figure 3-15, the temperature differences between the simulation results and the experimental data are generally within 10°C. The deviation from prediction is <2% in the worst case. Note that (a) there was no brazing between the center heat transfer pipe and the graphite foam and (b) the simulations were conducted with no gap between the center heat transfer pipe and the PCM–graphite foam composite. *Based on the results of the comparisons, the influence of the brazing between the center heat transfer pipe and the graphite foam on the overall thermal performance is insignificant.*

Figure 3-14. Temperature comparison for charging tests

Figure 3-15. Temperature comparison for discharging tests

4. Selection of Inexpensive Alloys for LHTES System

For an actual LHTES system with an s-CO₂ power cycle operating at higher temperatures, it is expected that the current alloys such as Incoloy 800H used for the lower temperature LHTES systems would not be applicable. To identify and establish potential alloys for LHTES systems (HTF pipe and containment module), a detailed literature search was conducted.

4.1. Material Survey

Several key properties of the potential alloys were identified:

- High-temperature yield stress;
- Corrosion properties, in the presence of PCM and s-CO₂;
- Oxidation resistance;
- Weldability;
- Thermal conductivity;
- Allowable stresses at elevated temperatures; and
- Current availability of the alloys.

Based on these criteria, several alloys were surveyed; the comparisons are listed in Table 4-1. Based on the quoted properties, a relative value ranging from 1 to 10 (10 being the best) was assigned for each alloy. In some cases, if the value was not found, a midpoint value was assigned. Table 4-1 shows that there several alloys such as Haynes 230 and 740H and Inconel 617 and 625 rank at the top.

Table 4-1. Literature survey of the properties of various high-temperature alloys

Property	Inconel 718	Inconel 617	Inconel 625	Sandvik Sanicro 25 (developed for use up to 700°C)	Haynes 282	Haynes 230	Special metal 740H
Composition	Ni, 55 Cr, 20 Nb+Ta, 5	Ni, 44 Cr, 22 Mo, 9 Al, 1.0	Ni, 58 Cr, 22 Nb, 4	Ni, 25 Cr, 22 W, 4 Cu, 3	Ni, 57 Cr, 20	Ni, 57 Cr, 20	Ni, 45 Co, 25 Cr, 25 Nb, 1.5 Al, 1.4
Yield stress (700–900°C)	340 MPa at 871°C 8 ^a	300 MPa at ~800°C 7	6	355 MPa at 800°C 8	~450 MPa at 871°C 9	340 MPa at ~800°C 8	594 MPa at 800°C 10
Corrosion with sCO ₂	5 ^b	750°C/2,900 psi s-CO ₂ , CR = 3 µg/mm ² after 1 000 hours 7	750°C/2,900 psi s-CO ₂ , CR = 1.5 µg/mm ² after 1,000 hours 9	5 ^b	750°C/2,900 psi s-CO ₂ , CR = 5 µg/mm ² after 1,000 hours 5	750°C/2,900 psi s-CO ₂ , CR = 1 µg/mm ² after 1,000 hours 10	750°C/2,900 psi s-CO ₂ , CR, =,3 µg/mm ² after 1,000 h 7
Oxidation resistance	6	6	5	7	9	9	8
Weldability	GTAW ^c 718 filler 7	7	Conventional welding/ electrode welding 9	GTAW 617 filler 7	GTAW/GMAW ^d 7	GTAW/GMAW 7	GTAW 740H filler 7
Thermal conductivity (at 800°C)	24 W/mK 8	23 W/mK 8	20-22 W/mK 7	25 W/mK 8	26 W/mK 8	24 W/mK 8	22 W/mK 8
ASME maximum		~5,000 psi at 800°C	Poor	4,900 psi at 760°C	No data	1,800/6,700 psi at 871/760°C	5,000 psi at 800 °C

allowable stress (at ~900°C)	8	9	6	6	Since better creep properties than 230 9	8	8
Available in dimensions required	0	5 ^b	9	0	0	0	0
Total score	42	49	51	41	47	50	49

^a Ranking 1–10, with 10 being the best.

^b Data were not found and the rating was assigned arbitrarily.

^c GTAW = gas tungsten-arc welding.

^d GMAW = gas metal-arc welding.

4.2. Corrosion Test Approach

Chloride salts have a high heat of fusion and hence are excellent PCMs for thermal energy storage. Argonne, with support from DOE's SunShot Initiative, has built and demonstrated a LHTES system. The performance testing showed that the LHTES system meets the targets. However, there are issues with the long-term interactions of the chloride salts (e.g., MgCl₂) with the metal components (containment and HTF pipes) in a realistic environment of the storage system. Previous work by Argonne and Arizona State University has shown that removing moisture and oxygen from the PCM salts can mitigate alloy corrosion [28]. However, the long-term corrosion rates of potential alloys have not been established in a realistic LHTES system environment of the Argonne storage technology in which the PCM is contained in the graphite foam pores and has minimal contact with the alloys.

The objective of this study was to quantify corrosion rates/depths of several alloys due to chloride (MgCl₂) PCM. This study established the long-term reliability of the LHTES system and validated the corrosion mitigation approaches: (a) argon degassing/cleaning of PCM salt prior to infiltration, (b) containment of PCM within the pores of the graphite foam, and (c) process steps used in the construction of the LHTES system. The approach was to evaluate the corrosion rates of the various alloys in carefully fabricated modules simulating realistic environment conditions. The results of this investigation will greatly aid in convincing industrial end users of the viability of the technology.

4.2.1. Metal Alloy Samples and Corrosion Test Modules

Based on the criteria of the yield stress, corrosion with s-CO₂, oxidation resistance, allowable stresses at elevated temperatures, current availability of the alloys, and so forth, several alloys were surveyed, as discussed in section 4.1. Based on the literature study, it was decided to evaluate Inconel 625 and Haynes 230 alloys for corrosion. Further, it was decided to also determine the effects of the PCM on the containment materials, that is, low-cost SS 304. Three alloys were investigated, namely, SS 304, Inconel 625, and Haynes 230. Alloys Inconel 625 and Haynes 230 are candidates for HTF piping for s-CO₂ power cycles, and SS 304 is an inexpensive containment material being used for the LHTES system.

To simulate the typical PCM-alloy physical interaction, disk-shaped alloy samples (Φ3 mm × 3/16 in. for SS 304, Φ3 mm × 3 mm for Inconel 625, and Φ3 mm × 4 mm for Haynes

230) were embedded in the graphite foam at two different depths, one close to the bottom and another near the top. This was done to determine whether there were any differences in the moisture/oxygen pickup by the PCM after infiltration. Six samples of each alloy were placed at the two depths, as shown in Figure 4-1, with three samples of each alloy for each depth.

Figure 4-1. Schematic diagrams of test modules for corrosion studies

The overall design of the corrosion test modules is shown in Figure 4-1. The container cylinders, bottom plates, top plates, and graphite foams were machined according to the design. The metal alloy samples were machined to the dimensions using an electrical discharge machining (EDM) technique. Then the container bottom plates were welded to the container cylinders, followed by a brazing process to ensure the graphite foams and the alloy metal samples remained in place during the MgCl₂ infiltration process. The fabrication process steps are shown in Figure 4-2.

Figure 4-2. Corrosion test module fabrication

4.2.2. Phase-Change Material Infiltration

Infiltration of the PCM (MgCl_2) into the graphite foam inside each corrosion test container was conducted by a vendor. To ensure the viability of the infiltration process, two trial runs with infiltration test containers were conducted, each run with two containers. The purpose of these runs was to ensure (a) the infiltration of the PCM in the graphite foam and (b) the complete filling in of the PCM around embedded metal samples, which would result in a realistic scenario of corrosion tests on high-temperature alloys.

For these tests, several test containers of SS 304 were fabricated. The graphite foam was machined into three separate disk-shaped pieces; in the bottom two sections slots were machined out to place the disks of the metal alloy samples. Subsequently, the graphite foam was brazed to the wall of the container to ensure the foam and the metal samples remained in place during the infiltration process.

Figure 4-3. Infiltration test containers

Each infiltration test container was weighed before and after infiltration to verify the amount of salt infiltrated into the graphite foam. After infiltration, each container was cut open to examine the graphite foams and embedded metals for infiltration results.

The first run did not generate the desired infiltration results. Based on analyses, the temperatures around the infiltration test containers were lower than those of the furnace controller. The salt was not completely melted during the run because the container temperatures were below the melting point of MgCl₂. Discussions between Argonne and the vendor resulted in a solution for this problem: adding a thermocouple near the infiltration test containers.

The second run was successful. As shown in Figure 4-4, the graphite foam was well infiltrated overall and around the embedded metal sample. Figure 4-4b shows the bottom of the container and the PCM is almost completely infiltrated. Figure 4-4c shows the metal disk sample that was placed in the foam. The PCM completely infiltrated the foam around the metal alloy. This gives confidence that embedded metal alloys can be used for conducting corrosion tests in a more realistic scenario.

(a) Container after infiltration

(b) Bottom part of container

(c) Embedded metal sample

(d) Graphite foam
filled with PCM

Figure 4-4. Infiltrated container and cut-open views

After the infiltration, the top plates of the containers were welded in a vacuum environment. In addition, there was a vacuum port on the top plate of each container to remove any residual moisture or oxygen in the system. The completed corrosion test modules are shown in Figure 4-5.

Figure 4-5. Completed corrosion test modules

4.3. Corrosion Experiments

4.3.1. Moisture and Oxygen Removal

For each container, a systematic moisture and oxygen removal process was followed: heating and holding the container at $>600^{\circ}\text{C}$; periodically vacuuming the container; and flushing the container with high-purity argon gas. Figure 4-6 shows the typical profile obtained during the cleaning procedure. Note that there are two regimes, at about 200°C and 450°C , at which the inside pressure rises rapidly, indicating emissions from minor decomposition of MgCl_2 to release HCl gas [29]. After the container vacuum pressure was stabilized for 96 hours at 600°C , the vacuum port was closed. Thereafter, the module was brought to room temperature; the vacuum line was isolated; and the assembly was sent to the machine shop to weld-shut the vacuum port. This welding was done under vacuum conditions to avoid moisture or oxygen pickup.

Figure 4-6. Temperature and pressure profiles during moisture removal process

4.3.2. Corrosion Testing

After welding, it was decided to add a cap to each test container in case there was a leak at the welding spot since the thickness of the weld was small. This is particularly important for the long-term tests. Figure 4-7 shows the corrosion test containers after installation of the cap and prior to the start of the corrosion testing. Figure 4-8 shows the containers placed in a precalibrated furnace for corrosion testing. Tests were conducted at 750°C for 100, 200, 500, and 1,000 hours. At each test interval, the furnace was cooled down to retrieve the test container from the furnace and then heated up to continue testing on the remaining containers.

Figure 4-7. Corrosion test containers with Inconel 625 caps to prevent salt leakage

Figure 4-8. Corrosion test containers in furnace for corrosion testing

4.3.3. Sample Cleaning

After corrosion testing, the containers were cut open and alloy samples extracted. Figure 4-9 shows the embedded samples after removal of the graphite foam and salt around

them. Since the salt and foam were fused to the sample surfaces, the following cleaning procedure was used:

- Sonicate the retrieved metal alloy samples in an isopropanol bath for 15 minutes.
- Wipe off any residue MgCl₂–graphite foam from the metal alloy samples.
- Sonicate the metal alloy samples in an isopropanol bath for 10 minutes.
- Dry the metal alloy samples in air.
- Sonicate the metal alloy samples in a clean isopropanol bath for 5 minutes.
- Dry the metal alloy samples in air.
- Dry the metal alloy samples at 60–70°C in a furnace for 20 minutes.

(a) Cut-open view

(b) Removal of salt on top

(c) Embedded alloy samples after removal of MgCl₂–graphite foam layer

Figure 4-9. Embedded alloy samples after corrosion testing

4.4. Experimental Results and Discussions

There are several methods for determining corrosion rates of alloys in molten salts, including (a) changes in weight and dimension with corrosion time, (b) change of corrosion zone/depth with time using microscopy, and (c) electrochemical techniques. In this study, the first two approaches were used for monitoring corrosion of the alloys. Estimating corrosion rates based on weight loss measurement can give erroneous results; hence absolute corrosion rates were not calculated. However, the measurements were used to identify trends in the corrosion process as a function of time.

4.4.1. Corrosion Rate Measurements Based on Weight Changes

After the retrieved alloy samples were cleaned, their masses were weighed, their dimensions measured, and the results compared to the original sample weights and dimensions. Figure 4-10 shows the change in weight (loss) for the three sets of alloys tested for 100, 200, 500, and 1,000 hours. For each alloy, samples placed in the upper and lower tiers of the container were combined to give an average value. Results reported are average for 4–6 samples. Sample weight losses were high for the initial 100-hour test for all samples. Weight losses for the subsequent 200- and 500-hour tests were of similar magnitudes. Further, for Inconel 625 and Haynes 230, the weight losses for 1,000 hours appear to be smaller but are within the scatter in the experimental data.

(a) SS 304

(b) Inconel 625

(c) Haynes 230

Figure 4-10. Weight loss of alloy samples as a function of corrosion time

Figure 4-11 shows the weight loss, on a percentage basis, of the three alloys when all the samples were combined and represented as an average. Results are consistent with the results in Figure 4-10.

(a) SS 304

(b) Inconel 625

(c) Haynes 230

Figure 4-11. Percentage weight loss of alloy samples as a function of corrosion time

Table 4-1 shows the weight loss rate normalized with the sample surface area. Two conclusions can be drawn from the data: (1) the weight loss rate decreases for each alloy with exposure time and (2) Haynes 230 and Inconel 625 appear to perform better than SS 304. Based on these results, estimated corrosion rates for SS 304, Inconel 625, and Haynes 230 are 95 $\mu\text{m/y}$, 9 $\mu\text{m/y}$, and 8 $\mu\text{m/y}$, respectively.

Table 4-1. Average weight loss normalized with surface area and exposure time

Test time	SS 304 (mg/cm ² -h)	Inconel 625 (mg/cm ² -h)	Haynes 230 (mg/cm ² -h)
100 hours	0.0513 ± 0.007	0.0149 ± 0.004	0.0118 ± 0.007
200 hours	0.0345 ± 0.003	0.0064 ± 0.002	0.0054 ± 0.002
500 hours	0.0144 ± 0.001	0.0032 ± 0.001	0.0025 ± 0.0004
1,000 hours	0.0085 ± 0.001	0.0008 ± 0.002	0.0008 ± 0.0002

In addition to weight, diameter changes in the samples were recorded. Diameter reductions are not quite as representative of corrosion as weight loss because of the fact that corrosion does not occur uniformly along the sample surface. Figure 4-12 shows the changes (reduction) in the diameter of the various samples as a function of corrosion exposure time. On a nominal basis, the diameter reductions follow the weight loss measurements, except for the 1,000-hour tests for Inconel 625 and Haynes 230.

Figure 4-12. Diameter reduction of alloy samples as a function of corrosion time

4.4.2. Corrosion Rate Measurements Based on Corrosion Depths

Scanning electron microscopy (SEM) and energy dispersive x-ray analysis (EDAX) tools were employed to estimate corrosion depths. Figures 4-13, 4-14, and 4-15 show typical SEM micrographs of corrosion on the sample surfaces of SS 304, Inconel 625, and Haynes 230 alloys for test intervals, respectively.

It is interesting to note the differences in the corrosion morphology in the three alloys. For SS 304 (Figure 4-13), corrosion at the surface was accompanied by cracks that extended inward. At 1,000 hours of exposure (Figure 4-13d), there was also segregation of chromium along the grain boundaries, which was later confirmed with EDAX.

Figures 4-14 and 4-15 show the SEM images for Inconel 625 and Haynes 230, respectively. For these samples, corrosion “affected zone” was well defined at the surface and accompanied with porosity, which eventually links up (Figure 4-15c), resulting in mass removal. Further, for all these alloys, after 1,000 hours of exposure, the segregation of chromium is observed at the grain boundaries and at the sample surfaces.

(a) 100 hours

(b) 200 hours

(c) 500 hours

(d) 1,000 hours

Figure 4-13. SEM images of corrosion progression in SS 304

(a) 100 hours

(b) 200 hours

(c) 500 hours

(d) 1,000 hours

Figure 4-14. SEM images of corrosion progression in Inconel 625

(a) 100 hours

(b) 200 hours

(c) 500 hours

(d) 1,000 hours

Figure 4-15. SEM images of corrosion progression in Haynes 230

For each sample evaluated, EDAX was conducted to profile the elemental distributions near the surface and along the corrosion depth. These analysis were conducted for all exposure times for each alloy. However, for brevity, data for 1,000-hour exposures are presented for the alloys in Figures 4-16, 4-17, and 4-18.

Figure 4-16. EDAX on SS 304 corrosion sample after 1,000-hour test

Figure 4-17. EDAX on Inconel 625 corrosion sample after 1,000-hour test

Figure 4-18. EDAX on Haynes 230 corrosion sample after 1,000-hour test

Figures 4-19, 4-20, and 4-21 show corrosion depth measurements at different exposure times for different samples. For each sample, 20–30 measurements of corrosion depths around the sample circumference were made and reported in the plots. Note that these measurements were from one of the flat ends of the sample. As shown in the plots, corrosion depth was quite constant over the four exposure times.

Figure 4-19. Corrosion depth measurements for SS 304

Figure 4-20. Corrosion depth measurements for Inconel 625

Figure 4-21. Corrosion depth measurements for Haynes 230

In addition, we included the depth at which chromium was segregated to determine how this region changed with exposure time. These measurements were made for the high-chromium alloys, Inconel 625 and Haynes 230, since it was readily visible in these alloys. Figure 4-22 shows the results from the same samples analyzed in Figures 4-20 and 4-21. As expected, the segregation depths are somewhat larger than the corrosion depths. For Haynes 230, this depth does not change significantly with time. This observation could indicate that the oxidation of chromium at the surface had diminished, and consequently, diffusion of chromium or segregation was limited. However, for Inconel 625, it increased with time, suggesting chromium diffusion was ongoing. This difference could be dependent on a variety of factors including grain size, alloy composition, and the like. Additional samples need to be analyzed to arrive at a firm conclusion.

Based on the corrosion tests, corrosion of the alloys (SS 304 and Haynes 230) is reduced with exposure times, indicating consumption of corrosion-causing species with the LHTES module. Although for Inconel 625 the corrosion depths increase with time, it

envisioned that at longer exposure times, the corrosion will taper off. The LHTES module is hermetically sealed, so it is expected that corrosion will eventually be below the desired levels. Further, these results validate the usage of relatively inexpensive SS 304 as a containment module for LHTES system.

Figure 4-22. Chromium segregation depth measurements for Inconel 625 and Haynes 230

5. Scale-up of Graphite Foam Process

Graphite foam is an integral part of the LHTES technology. Supplies of graphite foam with the desired properties are quite limited in the United States; the cost of the graphite foam is therefore high and affects the overall cost of the LHTES system. As part of this effort, a key task was to scale up the graphite foam production and ensure that the scaled-up foam met the proper requirements. The graphite foam scale-up activity was conducted primarily by Argonne's industry partner, Touchstone. The effort was divided into two phases: (a) producing laboratory-scale graphite foam and (b) scaling up the production to produce approximately 4,000 in.³ of the graphite foam.

5.1. Fabrication and Characterization of Initial Graphite Foam

Touchstone conducted the initial trials to produce laboratory-scale foam because, during the LPDP project, foam was provided by Koppers, Inc., and it was prudent to ensure that Touchstone could fabricate graphite foam similar to that from Koppers. The typical process involves first foaming the mesopitch carbon to make foam, followed by calcination and graphitization steps. Figure 5-1 shows the as-foamed pieces; some of the initial trial samples had cracks in them. Several of the as-foamed pieces were cut and loaded, along with an uncut as-foamed piece, into a small furnace for calcining (Figure 5-2). A total of eight pieces meeting the 6- × 4- × 4-in. desired size were fabricated. The three pieces with minimal cracking shown in Figure 5-3 resulted in finished pieces approximately 6 × 3.75 × 3.75 in. in size. Figure 5-4 shows all the foam samples after machining for graphitization.

The cracking was attributed to the large pans used for initial molding. It is believed that smaller pans result in improved temperature uniformity in the foam and reduce cracking. To avoid this, smaller pans were used that allowed for the production of individual pieces that meet the 6- × 4- × 4-in. size requirement.

Figure 5-1. As-foamed pieces

Figure 5-2. Foam pieces in calcining furnace

Figure 5-3. Foam pieces after calcining and machining

Based on the initial trials, a total of 11 blocks of nominal dimensions of 6 × 4 × 4 in. were fabricated. As shown in Figure 5-4, visual inspection showed the blocks are uniform in porosity, except at the top where there are large pores. Segregation of pores at the top is inherent in the foaming process, and this section is cut off, as shown in samples 2 and 6 in Figure 5-4. The nominal density and corresponding porosity of the foam blocks were measured; the results are presented in Table 5-1. The average porosity ranged between

81% and 85%, which is quite similar to the foam used in the LPDP project. SEM images (Figure 5-5) show the pore diameters in the range of 300–400 μm .

Figure 5-4. Graphite foam blocks fabricated by Touchstone

Table 5-1. Density and porosity measurements of fabricated graphite foam blocks

Block Number	Length (mm)	Width (mm)	Height (mm)]	Volume m ³	Mass (kg)	Density (kg/m ³)	Percentage Porosity
1	179.5	115.5	114.0	0.002363477	0.972	411.259	0.819
2	174.5	112.0	105.0	0.002052120	0.858	418.104	0.815
3	170.5	120.5	123.5	0.002537338	1.002	394.902	0.826
4	170.0	117.5	111.0	0.002217225	0.864	389.676	0.828
5	179.0	109.5	116.0	0.002273658	0.906	398.477	0.824
6	178.0	124.5	119.5	0.002648240	1.012	382.141	0.831
7	171.0	110.5	110.0	0.002078505	0.836	402.212	0.823
8	187.0	114.5	115.5	0.002473028	0.936	378.483	0.833
9	197.0	118.5	111.5	0.002602912	0.984	378.038	0.833
10	171.5	111.5	114.0	0.002179937	0.758	347.717	0.847
11	183.0	119.5	105.0	0.002296193	0.946	411.986	0.818

Figure 5-5. SEM of fabricated graphite foam

To ensure that the foam samples were sufficiently graphitized, x-ray diffraction was conducted on samples from two blocks (block 2 and block 6). The diffraction patterns in Figure 5-6 show the prominent peaks of graphite for both blocks. Further, there is some amorphous carbon in the foam as indicated by the broad peak at large d-spacing.

Figure 5-6. X-ray diffraction pattern for fabricated graphite foam samples

Thermal diffusivity and thermal conductivity measurements were conducted for samples from two graphite foam blocks (block 2 and block 6). The test sample preparation process is shown in Figure 5-7. First, the as-received graphite foam block (shown in Figure 5-7a) was cut along all six surfaces to form a smooth-surface block (shown in Figure 5-7b). Second, three thin plates (cut a, cut b, and cut c) were cut from three perpendicular surfaces of the block for representing its thermo-physical properties along three directions (shown in Figure 5-7c and Figure 5-7d). Third, four test samples (number 1, number 2, number 3, and number 4) were cored out from each plate (shown in Figure 5-7e). Fourth, all samples were slightly sanded along their circumferences for fitting into the sample holder for thermal diffusivity measurements. Finally, all samples were cleaned with compressed air to remove dust.

Figure 5-7. Sample preparation for thermal diffusivity measurements

Two samples for each cut direction of each block (a total of 12 samples) were used for thermal diffusivity measurements at room temperature. Three repeat measurements were conducted for each sample. Thermal conductivity together with deviation was calculated from the thermal diffusivity data. The results are summarized in Table 5-2. The data show (a) standard deviations for all measurements are generally small indicating the reliability of the measurements and (b) the thermal conductivity along three directions for all samples is generally within a reasonable range. Thus, the graphite foam fabrication process is repeatable and reliable.

Table 5-2. Experimental thermal diffusivity and calculated thermal conductivity of graphite foam blocks

Sample	Porosity	Thermal diffusivity (cm ² /s)		Density (kg/m ³)	Specific heat (kJ/kgK)	Thermal conductivity (W/mK)	
		Average	Deviation			Average	Deviation
2a2	0.76	0.8188	0.0021	553.3	0.7500	33.98	0.0864
2a4	0.76	1.1911	0.0081	554.3	0.7460	49.26	0.3335
2b2	0.83	2.2054	0.0181	399.1	0.7506	66.07	0.5430
2b4	0.83	1.6820	0.0030	396.1	0.7466	49.74	0.0895

2c1	0.72	1.1318	0.0064	653.2	0.7458	55.14	0.3113
2c4	0.71	1.0510	0.0044	661.7	0.7458	51.87	0.2155
6a2	0.83	1.5931	0.0032	398.4	0.7426	47.14	0.0932
6a4	0.83	2.0797	0.0304	382.7	0.7584	60.37	0.8837
6b1	0.83	1.5788	0.0033	403.5	0.7426	47.31	0.0989
6b2	0.82	1.5473	0.0071	410.1	0.7583	48.12	0.2210
6c2	0.63	0.7201	0.0073	856.6	0.7534	46.47	0.4721
6c3	0.63	0.5557	0.0255	844.5	0.7415	34.80	1.5979

Note that, for the prototype samples tested as part of the LPDP project, the graphite foam had a nominal porosity of 84% and an average thermal conductivity at room temperature of 55 W/mK. Thus the desired properties of the graphite foam were achieved.

5.2 Development and Characterization of Large-Scale Graphite Foam

5.2.1. Process Development

Based on the process described in section 5.1, Touchstone scaled up the process using large-scale calcination and graphitization furnaces. Figure 5-8 shows the fabricated foam samples. A total of 48 pieces with nominal dimensions of an 8-in. diameter and a 1.5-in. thickness were fabricated. Visual inspection showed the blocks were quite uniform in porosity with slight differences on the top and the bottom. These graphite foam blocks can be used to form a larger graphite foam structure. Figure 5.9 shows that 26 pieces were stacked together to form a 1-m-high graphite foam structure. This is sufficient to produce one subsystem-level module.

(a) Top

(b) Bottom

Figure 5-8. Large graphite foam sample (8-in. diameter and 1.5-in. thickness)

Figure 5.9. Graphite foam pieces stacked to form a larger structure (8 in. × 39 in.)

5.2.2. Density and Porosity

The density, porosity, thermal diffusivity, thermal conductivity, and thermal expansion of the large graphite foam structure were characterized. The density and the porosity were characterized for two types of samples. First, the density and porosity were determined through the measured diameters, thicknesses, and masses of 10 as-received samples with 8-in. diameter and 1.5-in. thickness. The calculated results, listed in Table 5-3, show that the graphite foam samples are quite uniform with an average density of approximately 419 kg/m³ (standard deviation of 6.67 kg/m³) and an average porosity of approximately 81.4% (standard deviation of 0.3%), which is quite similar to the small graphite foam.

Table 5-3. Density measurements of fabricated large graphite foam samples

Result	Diameter (mm)	Thickness (mm)	Volume (m ³)	Mass (kg)	Density (kg/m ³)	Porosity
Average	203.2	38.1	1.2356×10^{-3}	0.518	419.41	0.814
Deviation	-	-	-	0.008	6.67	0.003

Second, the density and porosity were calculated from the measured diameters, thicknesses, and masses of 24 samples used for thermal diffusivity measurements. To reflect the differences in the top–bottom direction and the planar direction as well as the distance from the center of the large graphite foam, the samples were cut in both directions and at different distances from the center of the large graphite foam, shown in Figure 5-10. Note that the graphite foam showed different porosities on the top and on the bottom; the labels of the cut-out samples were for the condition in which the porosity of the top was less than that of the bottom. Samples from the small graphite foam and the

large graphite foam exhibit a similar pore structure, as shown in Figures 5-10c and 5-10d. The results are listed in Table 5-4. The average density is 440 kg/m³, and the average porosity is 80.5%.

(a) Sample labels

(b) Sample cutout

(c) Sample from small graphite foam

(d) Sample from large graphite foam

Figure 5-10. Samples cut out for thermal diffusivity measurements

Table 5-4. Density measurements on thermal diffusivity measurement samples

Sample		Diameter (mm)	Thickness (mm)	Volume (mm ³)	Mass (g)	Density (kg/m ³)	Porosity
Planar	Average	12.5325	8.1722	1008.10	0.4398	436.17	0.807
	Deviation	0.0863	0.1346	19.31	0.0165	11.93	0.005
Top–bottom	Average	12.5014	8.2997	1018.76	0.4517	443.39	0.804
	Deviation	0.0649	0.0559	10.12	0.0104	9.19	0.004
Overall	Average	12.5169	8.2360	1013.43	0.4457	439.78	0.805
	Deviation	0.0763	0.1200	16.03	0.0148	11.05	0.005

5.2.3. Thermal Diffusivity and Thermal Conductivity

The thermal diffusivity of the 24 samples was measured by using the Discovery Xenon Flash System from TA Instruments. Three repeat measurements were conducted on each sample. The thermal conductivity and its deviation were calculated from the thermal diffusivity data. The results are summarized in Table 5-5. The data show that (a) standard deviations for all measurements are generally small, indicating the reliability of the measurements; (b) changes along the distance from the center are generally insignificant; and (c) thermal diffusivity and thermal conductivity are much higher along the top–bottom direction than along the planar direction. These results indicate that the graphite foam fabrication process is repeatable and reliable. These trends are similar to those of small graphite foams [22, 30].

Table 5-5. Experimental thermal diffusivity and calculated thermal conductivity

Sample	Density (kg/m ³)	Specific heat (kJ/kgK)	Porosity	Thermal diffusivity (cm ² /s)		Thermal conductivity (W/mK)	
				Average	Deviation	Average	Deviation
Planar	436.17	0.741	0.807	1.0847	0.0945	35.886	2.637
Top–bottom	443.39	0.742	0.804	4.4777	0.3787	151.317	13.174

Thermal diffusivity as a function of temperature was also measured for six samples; three samples ($x_i y_o z_t$, $x_i y_m z_t$, and $x_i y_i z_t$) were along the planar direction and the other three samples ($z_t x_i y_i$, $z_t x_i y_m$, and $z_t x_i y_o$) were along the top–bottom direction. The results, plotted in Figure 5-11, show that thermal diffusivity and thermal conductivity decrease with an increase in temperature. However, the decreasing rates reduce with temperature and level out at high temperatures.

(a) Samples along planar direction

(b) Samples along top-bottom direction

Figure 5-11. Thermal diffusivity and thermal conductivity as a function of temperature

5.2.4. Thermal Expansion

The thermal expansion was measured in accordance with ASTM E228 by NETZSCH using a DIL 402C pushrod dilatometer [31]. The thermal expansion and the coefficient of thermal expansion (CTE) are plotted in Figures 5-12 and 5-13, respectively. The CTE reference temperature is 30°C, and the CTE values below 100°C are not reliable because of startup heating effects.

Figure 5-12. Thermal expansion as a function of temperature

Figure 5-13. Coefficient of thermal expansion as a function of temperature

Based on the scale-up effort, a total volume of 4,000 in.³ of graphite foam was produced, and the physical and thermal properties were measured to ensure the desired thermo-physical properties met the requirements.

6. Techno-economic Analysis

The purpose of this effort was to (a) conduct an extensive analysis of the major components of the LHTES system, (b) estimate the range of storage costs, and (c) compare them with the DOE's target of \$15/kW_{th} and the current baseline for the power tower system using sensible heat storage. Subsequently, the potential areas of cost reductions and pathways to achieve these reductions could be identified.

The approach was similar to that used for the two-tank power tower system [23]. The key costs were identified to obtain a baseline cost of the LHTES system. A sensitivity analysis was conducted to identify the cost component with the greatest impact on overall LHTES storage costs. Finally, recommendations and solutions for mitigating the costs of the specific component to meet the overall target cost for the TES system are presented.

6.1. Baseline Storage System

The approach to the cost analysis of a full-scale LHTES was similar to the one used for the two-tank molten salt sensible heat thermal energy storage system (SHTES) [23]. The two-tank as the base case is a direct system; it is schematically shown in Figure 6-1 for a power tower CSP plant. The key assumptions for the cost analysis were as follows:

- Storage system for a 100 MWe for 6 hours;
- High-temperature tank, 565°C, and low-temperature tank, 293°C; $\Delta T \sim 272^\circ\text{C}$;
- High-temperature tank material, 321 stainless steel; low-temperature tank, carbon steel;
- Salt, nitrate salt mixture, cost \$1.23/kg;
- Electrical and instrumentation, 7% of total tank costs;
- Piping, valves, and fittings, 3% of total tank costs;
- Sales tax, 4%; and
- Contingency, 7%.

Figure 6-1. Various subsystems for a power tower-based CSP plant [32]

Table 6-1 shows the cost analysis for the current two-tank SHTES system [33]. Note that the pump costs were not included in the storage system cost analysis. According to the cost analysis, the storage cost is approximately \$26/kW_{th}. Note that the SHTES system presented here as the baseline is for 6-hour storage, and the storage system is compatible to the steam Rankine power cycle only.

Table 6-1. Cost analysis for direct, two-tank SHTES system (base case) [33]

Component	Materials (\$/kW _{th})	Installation (\$/kW _{th})	Total cost (\$/kW _{th})
High-temperature tank, stainless steel	5.20	1.84	7.04
Low-temperature tank, carbon steel	1.30	1.84	3.14
Tank supports, foundations, and site work	1.10	1.55	2.65
Storage medium	11.74	0.36	12.10
Electrical and instrumentation	0.46	0.43	0.90
Piping, valves, and fittings	0.20	0.18	0.38
Total	19.99	6.20	26.21

Note that the cost analysis is for a lower storage temperature range. In the recent Gen3 Demonstration Roadmap [34] storage costs for a two-tank system compatible with an s-CO₂ power cycle are estimated to be ~\$58/kW_{th}.

6.2. Cost Analysis Approach

The approach for conducting the cost analysis is shown in Figure 6-2.

Figure 6-2. Cost analysis approach

6.2.1. Basic Assumptions

For the cost analysis, the CSP plant capacity was assumed to be 100 MWe with an 8-hour charging time and a 12-hour discharging time. Based on an efficiency of the s-CO₂ power cycle of approximately 50%, the LHTES system requires 200 MW_t (2,400 MWh) thermal energy storage capacity, which is equivalent to approximately 15,342 tons of the graphite foam–NaCl combination as the storage medium. The HTF pipes are inserted in the graphite foam–NaCl for heat exchange. The HTF outlet temperature from the storage system should be ≥720°C to achieve the temperature requirement (~700°C) of the s-CO₂ power cycle. Details of the assumptions are listed in Table 6-2. Note that several assumptions are the results of the extensive parametric study. These include effects of the variables, such as HTF pipe diameter and wall thickness, foam conductivity and porosity, module container dimensions, and the like, on the LHTES system performance (full utilization of the PCM, HTF outlet temperature requirement, exergetic efficiency target, and so on). For reference, the algorithm used for the optimization of the key variables with respect to cost is shown in Figure 2-3.

Table 6-2. Assumptions for the LHTES system cost analysis

Assumptions for the cost analyses:		
Thermal energy storage capacity	2400 MWht	
Storage time	12 hrs	
Modular container size:		
OD (cm)	122	(Standard 48 inch pipe)
Wall thickness (cm)	0.95 (Standard Schedule)	
Height (m)	10	
Modular container material	Stainless steel	
Total # of modular containers	724	
HTF pipe size:		
OD (cm)	6.03	(Standard 2 inch pipe)
Wall thickness (cm)	0.87 (Schedule 160)	
Height (m)	10	
HTF pipe material	Haynes 230/Inconel 625	
# of HTF pipe per modular container	5065	
Graphite foam:		
Open porosity	90%	
Thermal conductivity at room temperature: (W/m K)		
z direction	45	
xy direction	24	
Storage medium	NaCl	
Insulation thickness (mm)	81.28	

6.2.2. Cost Model for Single-Tank and Seven-Pipe Modular LHTES Systems

6.2.2.1. Key Components and Costs

The key components and costs for the LHTES system were identified as

- PCM salt (NaCl),
- Graphite foam,
- Welding,
- HTF pipes,
- Infiltration,
- Brazing,
- Stainless steel module containers, and
- Insulation material.

In addition, costs for electrical and instrumentation and piping/valves/fittings were included in the analysis. These costs were 7% and 3% of the tank material costs, respectively [33].

6.2.2.2. Pricing Survey

In this analysis single price points were used for each component of the LHTES system. However, to determine the range of cost, a survey was undertaken to determine the range of pricing available for each key component. Table 5-9 lists the values available in the literature, online quotes, or vendor quotes. The values in boldface are the midpoint unit costs used in the analysis to obtain LHTES system storage costs, as discussed below. It is recognized that the material for HTF pipes will be alloys Haynes 230 or Inconel 625 for a s-CO₂ power cycle; however, their bulk prices are not currently readily available. Therefore, for analysis purposes, we have used the bulk alloy pricing for Incoloy 800H. This assumption is reasonable since the composition of Incoloy 800H is similar to that of alloys Haynes 230 and Inconel 625.

Table 6-3. Unit prices of components of LHTES system

Components in LHTES system	Unit price	Source
NaCl (PCM)	\$0.04/kg	[35]
	\$0.07/kg	[36]
	\$0.35/kg	[37]
Graphite foam	\$4.3/kg	[38]
	\$10/kg	[39]
	\$20/kg	[40]
	\$30/kg	[41]
Welding	\$3.24/ft	[42]
	\$3.60/ft	[43]
	\$4.87	[43]
HTF pipes	\$2.00/kg (Incoloy 800H)	[44]
	\$5.80/kg (Incoloy 800H)	[45]
	\$9.00/kg (Incoloy 800H)	[46]
Infiltration ^a	\$500/module unit	
	\$1000/module unit	
	\$2000/module unit	
Brazing ^a	\$550/module unit	
Stainless steel pipes	\$2.40/kg	[47]
	\$3.00/kg	[48]
	\$4.00/kg	[49]
Insulation material	\$10.50/ft	[25, 33]

^aUnit prices are based on the current prices for the laboratory-scale LHTES prototypes.

6.2.2.3. Range of LHTES Storage Costs with Midpoint, Low, and High Pricing for the Components

Tables 6-4 and 6-5 illustrate the detailed cost breakdown for the seven-pipe LHTES modular system and the single-tank LHTES system, respectively. The LHTES system has a 12-hour storage capacity for a 100 MW_e power plant. The installation costs in the tables were calculated based on the unit installation prices used for the current SHTES system [33]. The differences in storage costs between the single-tank and the seven-pipe

modular system are minimal. Further, brazing of the HTF pipe to the graphite foam does not have a significant impact on overall costs.

Table 6-4. Cost analysis of the seven-pipe modular LHTES system using midpoint pricing

Component	No brazing			With brazing		
	Materials	Installation	Total cost	Materials	Installation	Total cost
1. Salt	\$941,290	\$0	\$941,290	\$941,290	\$0	\$941,290
2. Graphite Foam	\$21,710,000	\$0	\$21,710,000	\$21,710,000	\$0	\$21,710,000
3. Welding	\$88,158	\$0	\$88,158	\$88,158	\$0	\$88,158
4. Tank/modular containers	\$6,672,000	\$4,416,000	\$11,088,000	\$6,672,000	\$4,416,000	\$11,088,000
5. HTF pipes	\$5,113,021	\$0	\$5,113,021	\$5,113,021	\$0	\$5,113,021
6. Infiltration	\$3,620,000	\$0	\$3,620,000	\$3,620,000	\$0	\$3,620,000
7. Brazing	\$0	\$0	\$0	\$398,200	\$0	\$398,200
8. Foundation	\$887,698	\$0	\$887,698	\$887,698	\$0	\$887,698
9. Thermal insulation	\$232,930	\$0	\$232,930	\$232,930	\$0	\$232,930
10. Electrical and instrumentation	\$467,040	\$516,000	\$838,299	\$467,040	\$516,000	\$838,299
11. Piping, valves, and fittings	\$200,160	\$216,000	\$359,271	\$200,160	\$216,000	\$359,271
12. Total	\$39,932,298	\$5,148,000	\$44,878,668	\$40,330,498	\$5,148,000	\$45,276,868
13. LHTES unit cost (\$/kW_{th})	-	-	\$18.70/kW_{th}	-	-	\$18.87/kW_{th}

Table 6-5. Cost analysis of the single-tank LHTES system using mid-point pricing

Component	No brazing			With brazing		
	Materials	Installation	Total cost	Materials	Installation	Total cost
1. Salt	\$941,290	\$432,000	\$1,373,290	\$941,290	\$432,000	\$1,373,290
2. Graphite foam	\$21,710,000	\$0	\$21,710,000	\$21,710,000	\$0	\$21,710,000
3. Welding	\$25,265	\$0	\$25,265	\$25,265	\$0	\$25,265
4. Tank/modular containers	\$508,800	\$4,416,000	\$4,924,800	\$508,800	\$4,416,000	\$4,924,800
5. HTF pipes	\$5,113,021	\$4,416,000	\$9,529,021	\$5,113,021	\$4,416,000	\$9,529,021
6. Infiltration	\$3,620,000	\$0	\$3,620,000	\$3,620,000	\$0	\$3,620,000
7. Brazing	\$0	\$0	\$0	\$398,200	\$0	\$398,200
8. Foundation	\$887,698	\$1,860,000	\$2,747,698	\$887,698	\$1,860,000	\$2,747,698
9. Thermal insulation	\$232,930	\$0	\$232,930	\$232,930	\$0	\$232,930
10. Electrical and instrumentation	\$35,616	\$516,000	\$537,075	\$35,616	\$516,000	\$537,075
11. Piping, valves, and fittings	\$15,264	\$216,000	\$230,175	\$15,264	\$216,000	\$230,175
12. Total	\$33,089,884	\$11,856,000	\$44,930,254	\$33,488,084	\$11,856,000	\$45,328,454
13. LHTES unit cost (\$/kW_{th})	-	-	\$18.72/kW_{th}	-	-	\$18.89/kW_{th}

With all component costs considered, the lowest and highest storage costs are \$11/kW_{th} and \$43/kW_{th}, as shown in Tables 6-6 and 6-7, respectively.

Table 6-6. Cost analysis of the seven-pipe modular LHTES system using low-point pricing

Component	No brazing			With brazing		
	Materials	Installation	Total Cost	Materials	Installation	Total Cost
1. Salt	\$537,880	\$0	\$537,880	\$537,880	\$0	\$537,880
2. Graphite foam	\$9,335,300	\$0	\$9,335,300	\$9,335,300	\$0	\$9,335,300
3. Welding	\$79,431	\$0	\$79,431	\$79,431	\$0	\$79,431
4. Tank/modular containers	\$5,337,600	\$4,416,000	\$9,753,600	\$5,337,600	\$4,416,000	\$9,753,600
5. HTF pipes	\$1,136,227	\$0	\$1,136,227	\$1,136,227	\$0	\$1,136,227
6. Infiltration	\$3,620,000	\$0	\$3,620,000	\$3,620,000	\$0	\$3,620,000
7. Brazing	\$0	\$0	\$0	\$398,200	\$0	\$398,200
8. Foundation	\$887,698	\$0	\$887,698	\$887,698	\$0	\$887,698
9. Thermal insulation	\$232,930	\$0	\$232,930	\$232,930	\$0	\$232,930
10. Electrical and instrumentation	\$373,632	\$516,000	\$744,891	\$373,632	\$516,000	\$744,891
11. Piping, valves, and fittings	\$160,128	\$216,000	\$319,239	\$160,128	\$216,000	\$319,239
12. Total	\$21,700,826	\$5,148,000	\$26,647,196	\$22,099,026	\$5,148,000	\$27,045,396
13. LHTES unit cost (\$/kWh_{th})	-	-	\$11.10/kWh_{th}	-	-	\$11.27/kWh_{th}

Table 6-7. Cost analysis of the seven-pipe modular LHTES system using high-point pricing

Component	No brazing			With brazing		
	Materials	Installation	Total Cost	Materials	Installation	Total Cost
1. Salt	\$4,706,450	\$0	\$4,706,450	\$4,706,450	\$0	\$4,706,450
2. Graphite foam	\$65,130,000	\$0	\$65,130,000	\$65,130,000	\$0	\$65,130,000
3. Welding	\$119,259	\$0	\$119,259	\$119,259	\$0	\$119,259
4. Tank/modular containers	\$8,896,000	\$4,416,000	\$13,312,000	\$8,896,000	\$4,416,000	\$13,312,000
5. HTF pipes	\$5,113,021	\$0	\$5,113,021	\$5,113,021	\$0	\$5,113,021
6. Infiltration	\$10,860,000	\$0	\$10,860,000	\$10,860,000	\$0	\$10,860,000
7. Brazing	\$0	\$0	\$0	\$398,200	\$0	\$398,200
8. Foundation	\$887,698	\$0	\$887,698	\$887,698	\$0	\$887,698
9. Thermal insulation	\$232,930	\$0	\$232,930	\$232,930	\$0	\$232,930
10. Electrical and instrumentation	\$622,720	\$516,000	\$993,979	\$622,720	\$516,000	\$993,979
11. Piping, valves, and fittings	\$266,880	\$216,000	\$425,991	\$266,880	\$216,000	\$425,991
12. Total	\$96,834,958	\$5,148,000	\$101,781,328	\$97,233,158	\$5,148,000	\$102,179,528
13. LHTES unit cost (\$/kWh_{th})	-	-	\$42.41/kWh_{th}	-	-	\$42.57/kWh_{th}

6.2.3 Sensitivity Analysis

To evaluate the effects of the various key costs for the LHTES systems (single-tank system and seven-pipe modular system), a sensitivity analysis was conducted. In this analysis, while the baseline (midpoint) prices are kept constant, the price of one component was varied over the range as determined from the pricing survey to determine total LHTES system storage costs. Figures 6-3a–e show the variation (percentage from the baseline midpoint costs) in the LHTES system storage costs for the various

component costs: PCM, alloy, infiltration, welding, and graphite foam. As shown in the various plots, the overall system storage costs are most sensitive to the graphite foam costs and can range $\pm 60\%$ over the pricing survey costs for the foam. For other components the effects are quite minimal. *Thus, based on this analysis, the graphite foam cost is a key determinant of the final cost of the LHTES system storage.* Finally, there are no significant differences in the sensitivity analysis between the single-tank and seven-pipe modular designs.

(a)

(b)

(c)

(d)

(e)

Figure 6-3. Sensitivity analysis of costs of various components

6.2.4. Cost Analysis as a Function of Graphite Foam Conductivity

Based on the earlier analysis, it is clear that the graphite foam price is the key determinant of the overall cost of the LHTES. The price of the graphite foam is related to the precursor material used, graphitization costs, machining, and minimization of the waste foam. Some of these factors determine the final thermal performance of the graphite foam. Hence, it is important to determine the optimum performance of the graphite foam in relation to its price and the resulting LHTES costs. Several simulations were conducted for the seven-pipe modular LHTES system for graphite conductivities of 10, 20, 45, and 150 W/mK. For each case, the LHTES system storage capacity was taken as 12 hours with s-CO₂ as the working fluid in the CSP plant. The 100-MW_e s-CO₂ power turbine is applied to the power plant. Subsequently, the number of HTF pipes, the number of modules needed, and so on were determined and used in the cost model to determine the LHTES storage costs.

Tables 6-8 to 6-11 show the costs for the LHTES systems with graphite foam thermal conductivities of 10, 20, 45, and 150 W/mK, respectively. The prices for the various components used here are the midpoint prices listed in Table 6-3. Also, the costs are given for systems with and without brazing. It is clear that as the thermal conductivity of the foam increases, there is a decrease in overall system costs. However, an increase in the thermal conductivity from 45 to 150 W/mK does not decrease the system cost because the amount of the graphite foam needed is more or less fixed based on the amount of PCM needed and the heat transfer does not contribute much toward reducing the total alloy components. Thus, a graphite foam with a thermal conductivity ~20 W/mK should be sufficient.

Table 6-8. LHTES unit cost with graphite foam thermal conductivity of 10 W/mK

Component	No brazing			With brazing		
	Materials	Installation	Total cost	Materials	Installation	Total cost
1. Salt	\$867,230	\$0	\$867,230	\$867,230	\$0	\$867,230
2. Graphite foam	\$20,000,000	\$0	\$20,000,000	\$20,000,000	\$0	\$20,000,000
3. Welding	\$113,281	\$0	\$113,281	\$113,281	\$0	\$113,281
4. Modular containers	\$7,882,929	\$4,416,000	\$12,298,929	\$7,882,929	\$4,416,000	\$12,298,929
5. HTF pipes	\$8,064,744	\$0	\$8,064,744	\$8,064,744	\$0	\$8,064,744
6. Infiltration	\$5,710,000	\$0	\$5,710,000	\$5,710,000	\$0	\$5,710,000
7. Brazing	\$0	\$0	\$0	\$628,100	\$0	\$628,100
8. Foundation	\$691,440	\$0	\$691,440	\$691,440	\$0	\$691,440
9. Thermal insulation	\$195,279	\$0	\$195,279	\$195,279	\$0	\$195,279
10. Electrical and instrumentation	\$551,805	\$516,000	\$909,326	\$551,805	\$516,000	\$909,326
11. Piping, valves, and fittings	\$236,488	\$216,000	\$389,711	\$236,488	\$216,000	\$389,711
12. Total	\$44,313,195	\$5,148,000	\$49,239,939	\$44,941,295	\$5,148,000	\$49,868,039
13. LHTES unit cost (\$/kWh_{th})	-	-	\$20.52/kWh_{th}	-	-	\$20.78/kWh_{th}

Table 6-9. LHTES unit cost with graphite foam thermal conductivity of 20 W/mK

Component	No brazing			With brazing		
	Materials	Installation	Total cost	Materials	Installation	Total cost
1. Salt	\$867,230	\$0	\$867,230	\$867,230	\$0	\$867,230
2. Graphite foam	\$20,000,000	\$0	\$20,000,000	\$20,000,000	\$0	\$20,000,000
3. Welding	\$90,504	\$0	\$90,504	\$90,504	\$0	\$90,504
4. Tank/modular Containers	\$6,652,868	\$4,416,000	\$11,068,868	\$6,652,868	\$4,416,000	\$11,068,868
5. HTF pipes	\$5,785,336	\$0	\$5,785,336	\$5,785,336	\$0	\$5,785,336
6. Infiltration	\$4,095,000	\$0	\$4,095,000	\$4,095,000	\$0	\$4,095,000
7. Brazing	\$0	\$0	\$0	\$450,450	\$0	\$450,450
8. Foundation	\$674,845	\$0	\$674,845	\$674,845	\$0	\$674,845
9. Thermal insulation	\$190,775	\$0	\$190,775	\$190,775	\$0	\$190,775
10. Electrical and instrumentation	\$465,701	\$516,000	\$822,060	\$465,701	\$516,000	\$822,060
11. Piping, valves, and fittings	\$199,586	\$216,000	\$352,311	\$199,586	\$216,000	\$352,311
12. Total	\$39,021,844	\$5,148,000	\$43,946,929	\$39,472,294	\$5,148,000	\$44,397,379
13. LHTES unit cost (\$/kWh_{th})	-	-	\$18.31/kWh_{th}	-	-	\$18.50/kWh_{th}

Table 6-10. LHTES unit cost with graphite foam thermal conductivity of 45 W/mK

Component	No brazing			With brazing		
	Materials	Installation	Total cost	Materials	Installation	Total cost
1. Salt	\$867,230	\$0	\$867,230	\$867,230	\$0	\$867,230
2. Graphite foam	\$20,000,000	\$0	\$20,000,000	\$20,000,000	\$0	\$20,000,000
3. Welding	\$75,892	\$0	\$75,892	\$75,892	\$0	\$75,892
4. Tank/modular Containers	\$5,831,442	\$4,416,000	\$10,247,442	\$5,831,442	\$4,416,000	\$10,247,442
5. HTF pipes	\$4,400,328	\$0	\$4,400,328	\$4,400,328	\$0	\$4,400,328
6. Infiltration	\$3,115,000	\$0	\$3,115,000	\$3,115,000	\$0	\$3,115,000
7. Brazing	\$0	\$0	\$0	\$342,650	\$0	\$342,650
8. Foundation	\$670,236	\$0	\$670,236	\$670,236	\$0	\$670,236
9. Thermal insulation	\$189,882	\$0	\$189,882	\$189,882	\$0	\$189,882
10. Electrical and instrumentation	\$408,201	\$516,000	\$764,237	\$408,201	\$516,000	\$764,237
11. Piping, valves, and fittings	\$174,943	\$216,000	\$327,530	\$174,943	\$216,000	\$327,530
12. Total	\$35,733,153	\$5,148,000	\$40,657,777	\$36,075,803	\$5,148,000	\$41,000,427
13. LHTES unit cost (\$/kWh _{th})	-	-	\$16.94	-	-	\$17.08

Table 6-11. LHTES unit cost with graphite foam thermal conductivity of 150 W/mK

Component	No brazing			With brazing		
	Materials	Installation	Total cost	Materials	Installation	Total cost
1. Salt	\$867,230	\$0	\$867,230	\$867,230	\$0	\$867,230
2. Graphite foam	\$20,000,000	\$0	\$20,000,000	\$20,000,000	\$0	\$20,000,000
3. Welding	\$75,283	\$0	\$75,283	\$75,283	\$0	\$75,283
4. Tank/modular Containers	\$5,784,640	\$4,416,000	\$10,200,640	\$5,784,640	\$4,416,000	\$10,200,640
5. HTF pipes	\$4,362,977	\$0	\$4,362,977	\$4,362,977	\$0	\$4,362,977
6. Infiltration	\$3,090,000	\$0	\$3,090,000	\$3,090,000	\$0	\$3,090,000
7. Brazing	\$0	\$0	\$0	\$339,900	\$0	\$339,900
8. Foundation	\$665,165	\$0	\$665,165	\$665,165	\$0	\$665,165
9. Thermal insulation	\$188,990	\$0	\$188,990	\$188,990	\$0	\$188,990
10. Electrical and instrumentation	\$404,925	\$516,000	\$760,606	\$404,925	\$516,000	\$760,606
11. Piping, valves, and fittings	\$173,539	\$216,000	\$325,974	\$173,539	\$216,000	\$325,974
12. Total	\$35,612,749	\$5,148,000	\$40,536,866	\$35,952,649	\$5,148,000	\$40,876,766
13. LHTES unit cost (\$/kWh _{th})	-	-	\$16.89/kWh _{th}	-	-	\$17.03/kWh _{th}

Further, the same analysis was conducted in which the high and low prices of each LHTES component were considered. Figure 6.4 shows the LHTES cost variations as a function of the graphite foam thermal conductivity at low, medium, and high price points of the various components. It is clear from this analysis that at mid and low price points and a thermal conductivity less than 45 W/mK, there is a high potential for meeting the DOE's target storage costs using the developed LHTES technology.

Figure 6.4. LHTES unit cost versus graphite foam thermal conductivity

Therefore, it is not necessary to pursue extremely high thermal conductivity graphite foam. A graphite foam thermal conductivity of 20 W/mK would be enough to achieve the heat transfer performance in the storage system, which can help reduce the graphite foam manufacturing price.

6.2.5 Foam Costs and Target Pricing

Based on the techno-economic analysis, several road maps will need to be developed to reduce the cost to the \$10–\$15/kg range with the thermal conductivity of ~20 W/mK to make the LHTES system meet the DOE's storage cost target of \$15/KW_{th}. This work has demonstrated that a low-cost production route is feasible for producing graphitic foam from mesophase pitch. The large-scale manufacturing route adopted by Touchstone has the potential to reduce the cost of graphitic foam by up to 50% compared to the baseline when the project first began. Pricing for graphite foam as of the end of this effort was 33% less than the baseline price. As the product expands further into the marketplace, the current pricing may be revisited and adjusted based on higher volume sales.

In fabrication of the graphite foam, the main cost is the precursor mesopitch material. Mesopitch carbon provides high-quality graphite foam. As discussed previously, it is not necessary to use extremely high-thermal-conductivity graphite foam. Thermal simulations and techno-economic analyses have shown that thermal conductivity in the range of 20–25 W/mK would be acceptable to meet performance targets. Thus, this provides an opportunity to use less costly precursor materials for the fabrication of the graphite foam. Touchstone will be exploring this option based on its inexpensive CFoam® (carbon foam) products. In these products the foam is manufactured by using a majority of coal tar-based precursor material blended with mesopitch carbon. Coal tar-based precursor is two orders of magnitude cheaper than mesopitch. By tailoring the processing, it is expected that thermal conductivities up to 25 W/mK can be achieved. These carbon-based foams are significantly cheaper than foams with pure graphitic structures. Touchstone expects to further optimize the low-cost foam and scale the process for large-scale manufacturing.

7. Summary

1. The heat transfer performance of the LHTES system was improved with graphite foam, compared to that without graphite foam, as established by numerical simulations for a full-scale 8-hour charging and 12-hour discharging thermal energy storage system for CSP plant applications. The results showed that a reduction factor of 12 on the number of HTF pipes could be achieved.
2. The optimization of the modular LHTES system design was conducted through parametric studies. The results showed that (a) the open porosity of graphite foam should be >84%; (b) the optimum HTF pipe size should be 2 in. with a schedule 160 wall thickness considering the combined effects of exergy efficiency, high operating pressure (200 bar) of s-CO₂, and costs; (c) the interface gap thickness between the HTF pipe and the graphite foam should be less than 0.5 mm based on the system heat transfer performance; and (d) the optimum modular container size would be 48-in. pipe (~1.2 m outer diameter) with a standard wall thickness (0.375 in.).
3. The advantages of the modular LHTES system over the current fin-structured tube-in-shell thermal energy storage system were investigated. The results showed that (a) the heat transfer rate in the modular graphite foam-PCM system was approximately 3 times higher than that in the fin-structured tube-in-shell PCM system and (b) the energetic efficiency and the exergy efficiency of the modular graphite foam-PCM system were 100% and 96.7%, respectively, while those of the fin-structured tube-in-shell PCM system were 80% and 77.3%, respectively.
4. A detailed efficiency analysis was conducted for the modular LHTES system. The results showed that after incorporating the heat loss, the energetic and exergy efficiencies were greater than 99% and 95%, respectively.
5. Based on the thermal performance data from the experimental tests of the inexpensive prototype with no brazing between the center HTF pipe and the graphite foam, the temperature profiles followed the similar heat storage/release trends as those of the previous prototypes with brazing between the center HTF pipe and the graphite foam.
6. The thermal performance results of the experimental tests and the numerical simulations agreed very well. The temperature differences between the experimental tests and the numerical simulations were within 5°C and 10°C for charging and discharging, respectively.
7. Based on comparisons of the prototypes with and without brazing between the center HTF pipe and the graphite foam, it was concluded that eliminating the brazing would not cause significant thermal performance degradation.
8. To down-select a low-cost containment alloy, corrosion effects of MgCl₂ on the SS 304, Inconel 625, and Haynes 230 alloys were investigated through embedded

metal samples in the graphite foam–MgCl₂ medium heated to and kept at 750°C for 100, 200, 500, and 1,000 hours. Experimental testing was conducted in a controlled environment after a moisture/oxygen removal process.

9. The corrosion rates were determined for the testing alloys by examining (a) changes in weight and dimension with exposure time and (b) changes in corrosion zone and depth with exposure time with microscopy. The results showed that (a) the weight loss rate for each alloy decreased with exposure time and (b) Haynes 230 and Inconel 625 appeared to be better performers than SS 304. The estimated corrosion rates for SS 304, Inconel 625, and Haynes 230 were 95, 9, and 8 μm/y, respectively.
10. In addition, the depth at which chromium was segregated was also measured as a function of exposure time for the high-chromium alloys Inconel 625 and Haynes 230. As expected, the segregation depths were somewhat larger than the corrosion depths. Furthermore, depth did not change significantly with exposure time for Haynes 230, while it increased with exposure time for Inconel 625.
11. Extensive techno-economic analysis was conducted to estimate the cost range of the modular LHTES system. The results showed that several road maps would need to be developed to reduce the graphite foam cost to a \$10–\$15/kg range with the thermal conductivity of ~20 W/mK to make the modular LHTES system meet the DOE's storage cost target of \$15/KW_{th}.
12. Large graphite foams were developed. Their thermo-physical properties were characterized. Overall, large graphite foams showed features similar to those of small graphite foams in density, porosity, thermal diffusivity, thermal conductivity, and thermal expansion as well as in CTE.
13. During the project period, preliminary technology-to-market and commercialization plans were developed, multiple technology-to-market activities were conducted, and media coverage was prepared.

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Appendix: Thermal Simulations of the LHTES System for Various HTFs

The thermal performance of the LHTES system (graphite foam plus MgCl₂ PCM) was considered using three different types of HTFs in the simulations: liquid sodium (Na), liquid chloride salt, and helium at 50 bar. The thermal properties of the three types of HTFs are displayed in Table A1.

Table A1. Thermophysical properties of HTFs [1–5]

HTF	Density (kg/m ³)	Specific heat (J/kgK)	Thermal conductivity (W/mK)	Dynamic viscosity (Pa·s)
Liquid Na	778.5	1260	59.7	0.00021
Liquid chloride salt (MgCl ₂ -NaCl eutectic) ^a	1680	1096	0.5	0.00136
Helium at 50 bar	2.46	5195	0.36	4.54 × 10 ⁻⁵

^a MgCl₂-NaCl eutectic (57% mole fraction NaCl with melting point of 444 °C [Zheng et al.])

The heat transfer performances of the LHTES system with various types of HTFs were simulated and compared: the HTF flows from the top of the modular container to the bottom during the charging process, and from the bottom to the top during the discharging process. The CSP plant capacity was 100 MW_e with 12-hour storage capacity and thermal to electricity conversion efficiency of ~50%. Therefore, the required LHTES system storage capacity was 200 MW_{th} with an 8-hour charging time and a 12-hour discharging time. The heat loss of the storage system was assumed to be approximately 1%. The HTF inlet temperature was maintained at 750°C for the charging process and at 520°C for the discharging process.

Tables A2 and A3 illustrate the charging and discharging conditions, the geometries, and the performances of the LHTES system with various types of HTFs. Both liquid Na and chloride salt had good heat transfer performances. The heat transfer performance of helium gas is not as good as that of liquid HTFs. For liquid Na and chloride salt, approximately 1,095 modular containers with about 7,650–7,690 HTF pipes were needed to achieve a storage capacity of 200-MW_{th}. For the helium gas, over 2.5 times more modules and HTF pipes were required for the full-scale LHTES system compared to the liquid HTFs, as illustrated in Table A3, because of the relatively poor heat transfer performance of the gas HTF. Furthermore, the round-trip exergy efficiencies of the graphite foam–MgCl₂ LHTES system were more than 95% for different types of HTFs based on the simulations because of the high thermal conductivity of the graphite foam component.

Table A2. Charging and discharging conditions of the LHTES system with various types of HTFs

Condition	Charging Process			Discharging Process		
	Liquid Na	Liquid chloride salt	Helium at 50 bar	Liquid Na	Liquid chloride salt	Helium at 50 bar
Flow velocity, m/s	1	1	12	0.09	0.05	2.5
HTF inlet temperature, °C	750	750	750	520	520	520
HTF outlet temperature, °C	726.8	737.6	692.7	695.8	689.3	707.1

Table A3. Geometry and exergy efficiency of the LHTES system with various types of HTFs

	Liquid Na	Liquid chloride salt	Helium at 50 bar
Modular container ^a outer diameter, in.	28	28	18
Number of modules	1098	1093	2823
Number of HTF pipes	7686	7651	19761
Round-trip exergy efficiency of the LHTES system	95.3%	95.0%	96.2%

^a Modular container wall thickness: 0.375 in.

Figure A1 shows the locations of the melting or solidification fronts in the modules for different types of HTFs. Liquid Na and liquid chloride salt provided much better heat transfer than helium gas. At the same time point, more PCM was melted or solidified in the module with liquids as HTFs. Thus, the module diameter for the helium gas HTF was only 18 in., while it was 28 in. for liquid HTFs, as illustrated in Table 3. The single-module volume was much smaller for the helium gas HTF in order to achieve the completed melt/solidification inside, leading to more modules and HTF pipes required for the full-scale LHTES system.

Figure A1. Locations of the melting/solidification fronts in the modules using different types of HTFs: (a)–(c) melting fronts at 4 hours of the charging process using (a) liquid Na, (b) liquid chloride salt, and (c) 50-bar helium gas as the HTFs; (d)–(f) solidification fronts at 5 hours of the discharging process using (d) liquid Na, (e) liquid chloride salt, and (f) 50-bar helium gas as the HTFs

Figures A2 and A3 show the temperature distribution in the modules at various times during the charging and discharging processes, respectively. Liquid Na was considered as the HTF in the pipes. The figures display the temperature changes with time. For the charging process, the graphite foam–MgCl₂ composite temperature at the top of the module increased first, and then the heat spread down to the bottom because the HTF flowed from the top to the bottom. For the discharging process, the flow direction reversed. Therefore, the storage medium at the bottom of the module solidified first. The fluid flow direction was determined to make the molten PCM stay on the top of the module instead of the bottom to avoid the rapid increase of pressure inside the container because of the volume expansion of PCM during the phase change process.

Figure A2. The temperature distribution in the modular containers at various times during the charging process with liquid Na as the HTF: (a)–(c) the cross-section view of temperature at the middle of the module ($z = 5$ m) at various times, (a) 2 hours, (b) 4 hours, and (c) 6 hours; (d)–(f) the overall view of the modular temperature distribution at various times, (d) 2 hours, (e) 4 hours, and (f) 6 hours

Figure A3. The temperature distribution in the modular containers at various times during the discharging process with liquid Na as the HTF: (a)–(c) the cross-section view of temperature at the middle of the module ($z = 5$ m) at various times, (a) 2 hours, (b) 4 hours, and (c) 6 hours; (d)–(f) the overall view of the modular temperature distribution at various times, (d) 2 hours, (e) 4 hours, and (f) 6 hours

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