Development of Graphite Foam Infiltrated with MgCl₂ for a Latent Heat Based Thermal Energy Storage (LHTES) System

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Abstract

Thermal energy storage (TES) systems that are compatible with high temperature power cycles for concentrating solar power (CSP) require high temperature media for transporting and storing thermal energy. To that end, TES systems have been proposed based on the latent heat of fusion of the phase change materials (PCMs). However, PCMs have relatively low thermal conductivities. In this paper, use of high-thermal-conductivity graphite foam infiltrated with a PCM (MgCl_2) has been investigated as a potential TES system. Graphite foams with two porosities were infiltrated with MgCl_2 . The infiltrated composites were evaluated for density, heat of fusion, melting/freezing temperatures, and thermal diffusivities. Estimated thermal conductivities of MgCl_2 /graphite foam composites were significantly higher than those of MgCl_2 alone over the measured temperature range. Furthermore, heat of fusion, melting/freezing temperatures, and densities showed comparable values to those of pure MgCl_2 . Results of this study indicate that MgCl_2 /graphite foam composites show promise as storage media for a latent heat thermal energy storage system for CSP applications.

Keywords: latent thermal energy storage, concentrating solar power, phase change material, graphite foam, infiltration, magnesium chloride

1. Introduction

If concentrated solar power (CSP) is to be a viable technology for producing cost-effective grid electricity, thermal energy storage (TES) will be needed to generate electricity during nighttime and cloudy days and thereby improve the overall plant efficiency. With increased plant efficiency, the levelized cost of electricity would be reduced. One of the approaches for increasing plant efficiency is to deploy higher temperature power cycles, such as use of supercritical steam. The inlet temperatures for the heat transfer fluids (HTFs) for supercritical steam is 540-620 °C. This temperature range implies that the TES systems would need to operate such that during the system discharge the outlet temperature of the HTF is >650°C [1, 2].

Recently, various researchers have proposed TES systems for CSP based on the latent heat of phase change materials (PCMs). However, PCMs have relatively low thermal conductivity [3], which leads to long times to charge/discharge the storage system. In addition, PCMs are not fully utilized during the desired 8 hours of charging time. Therefore, several studies have focused on

enhancing the heat transfer characteristics of the PCMs. Finned tubes and heat pipes have been proposed to reduce the thermal resistance between the HTF and the PCM [4-13], while particles and metal foam structures with high thermal conductivities have been suggested to reduce the thermal resistance of the PCM itself [14-20].

Recent development of graphite foams with extremely high bulk thermal conductivities [21] has led to several investigations on using them for improving the thermal performance of low thermal conductivity materials [22-28]. Zhong et al. [24] showed that the thermal diffusivity of a paraffin/graphite foam composite was 570 times higher than that of pure paraffin wax. Warzoha et al. [25, 26] used a simple submersion technique combined with a vacuum infiltration technique to infiltrate the foam with paraffin wax. Their results showed that the vacuum infiltration technique was better as compared to the simple submersion technique, and it can fully infiltrate the foam with paraffin wax. Xiao and Zhang [27] compared the infiltration rates for the cases with and without vacuum assistance, and their results showed that the infiltration process with vacuum assistance was superior to that without it. Further, they reported that the thermal conductivity of the composite was higher compared to pure paraffin. The phase change behaviors of the composite were also investigated with a differential scanning calorimeter (DSC). The presence of the porous carbon foam did not affect the phase change behavior of pure paraffin wax.

Recently, Sedeh and Khodadadi [28] investigated the effective thermal conductivity of composites of graphite foam infiltrated with cyclohexane as the PCM. Their results indicated an improvement in the effective thermal conductivity of composites compared to that of the pure PCM at temperatures up to 70°C. Their numerical simulation and experimental results were in good agreement. They also found that the role of natural convection within the pores was negligible.

There are only a few studies on the use of high conductivity foams with high melting point PCMs [29-31]. Kim et al. [29] conducted a parametric study of latent heat thermal energy storage (LHTES) systems using graphite foams and high temperature PCMs. They found that the use of the graphite foam and the turbulent flow could significantly reduce the number of heat transfer fluid pipes. Zhao et al. [30] investigated the exergy efficiency in LHTES systems using the MgCl_2 -graphite foam composite. They found that the graphite foam could significantly improve the heat transfer performance as well as the energy efficiency in the LHTES system. Recently, Jianfeng et al. [31] developed a high temperature energy storage material using silicon carbide (SiC) ceramic foam as matrix and Na_2SO_4 as phase change material with melting point of 884 °C. In this work, sodium sulfate was infiltrated into the silicon carbide foam via the melt infiltration method. The resulting composite thermal conductivity was found to be ~6 W/m-K, and the thermal energy storage density was 161 kJ/kg as the composite was cycled between 800 °C and 900 °C. Further, thermal cycling did not degrade the composite performance.

The focus of this work is to develop a graphite foam/PCM composite that could be used in an LHTES system that will be compatible with a supercritical steam power cycle. In this regard, we selected magnesium chloride (MgCl_2) as the PCM, which has a melting point of 714°C, and infiltrated it into the graphite foam preform. Various physical and thermal characterizations were performed on the fabricated composite to validate its use for the LHTES system.

2. Experimental Procedure

(a) $MgCl_2$ PCM

In the present study, magnesium chloride ($MgCl_2$) was chosen as the PCM for the infiltration experiments because of its melting temperature range, high volumetric heat of fusion, small under-cooling temperature, and low vapor pressure [32]. The physical and thermal properties of $MgCl_2$ are summarized in Table 1.

For the experimental work, $MgCl_2$ was obtained from a commercial source (98% purity, ACROS Organics). The as-received $MgCl_2$ powders and the foam/PCM composites were characterized for weight loss as a function of temperature, melting point, and heat of fusion by means of a differential scanning calorimeter-thermogravimetric analyzer (DSC-TGA, Model SDT-Q600, TA Instruments).

(b) Graphite foam

Two kinds of commercial graphite foams that have different porosities were used in the present study: POCO-HTC (high density) and Graftech (low density). From here on, the POCO-HTC and Graftech foams will be referred to as high density (HD) and low density (LD) foams, respectively. The as-received graphite foams were characterized for density (gas pycnometer, Model Ultrapyc 1200e, Quantachrome Instruments). The microstructure of the graphite foam samples, both as-received and after infiltration, was examined by scanning electron microscopy (SEM) and energy dispersive x-ray analysis (EDX). Due to the processing of the graphite foam, its properties are anisotropic. They are the same in the x-y plane but different in the out-of-plane z direction, which is the rise direction of the foam. The manufacturer-provided thermal conductivities of the HD and LD foams are 245 W/m-K and 50 W/m-K in the z direction, and 70 W/m-K and 25 W/m-K in the x-y directions, respectively.

(c) Infiltration of graphite foam with $MgCl_2$

A pressure-assisted melt process was used to infiltrate $MgCl_2$ into the graphite foams. The graphite foam samples and $MgCl_2$ powder were placed in separate chambers, as shown schematically in Figure 1. The graphite foam chamber was evacuated while the PCM salt was heated to >800 °C in an argon gas cover. Once the salt was completely melted, the valve separating the two chambers was opened to allow the melt to infiltrate the graphite foam. In addition, gas pressure was used to ensure complete infiltration of the foam with PCM. Post infiltration, any excess PCM was gently wiped off. Typical dimensions of the infiltrated samples were 15.2 cm x 7.6 cm x 1.9 cm.

(d) Measurements of thermal diffusivity and thermal conductivity

Thermal conductivity is related to thermal diffusivity, density, and specific heat capacity at constant pressure as per the following equation:

$$k = \alpha \rho c_p \quad (1)$$

where k is thermal conductivity, α is thermal diffusivity, ρ is density, and c_p is specific heat capacity. Therefore, to determine the thermal conductivity of a material system, its thermal diffusivity, density, and specific heat need to be measured independently.

For this paper, thermal diffusivity was measured by applying an energy source on one face of the sample, and the temperature rise was recorded as a function of time. A commercial unit (Model DXF-900, TA Instrument) that employs a xenon flash was used for the thermal diffusivity measurements. These measurements provided accurate thermal transport properties of the baseline graphite foam and post-infiltration composite as a function of temperature.

The density and specific heat capacity of the composites were calculated from the following equations:

$$\rho_{composite} = \varphi_{foam}\rho_{PCM} + (1 - \varphi_{foam})\rho_{foam} \quad (2)$$

$$c_{p,composite} = \frac{\varphi_{foam}\rho_{PCM}c_{p,PCM} + (1 - \varphi_{foam})\rho_{foam}c_{p,foam}}{\rho_{composite}} \quad (3)$$

where φ_{foam} is porosity of the graphite foam. Density and specific heat capacity of PCM and foam were determined as a function of temperature from data in Ref. [32] and used for the calculation of the thermal conductivity of the composites with Eq. 1.

The thermal diffusivities of $MgCl_2$ /graphite foam composites were measured for both the z direction and x-y directions because graphite foams have different thermal conductivities for different orientations. In addition, the thermal diffusivities of graphite foams without $MgCl_2$ were measured to investigate the effect of infiltration of $MgCl_2$ on the thermal conductivity of the foam. Three samples were tested for each foam type to derive an average value with standard deviation. Dimension of all samples and experimental conditions are summarized in Table 2.

3. Results and Discussion

The microstructures of the as-received graphite foams are shown in Figure 2. As measured by a gas pycnometer, the HD foam has a density of $0.89 \pm 0.01 \text{ g/cm}^3$ and a porosity of $55.4 \pm 0.2\%$, while the LD foam has a nominal density of $0.23 \pm 0.01 \text{ g/cm}^3$ and a porosity of $90.7 \pm 0.3\%$. As provided by the manufacturer, nominal pores per inch were 16 and 35 for the HD and LD foams, respectively.

(a) Infiltration rate

Figure 3 shows the HD and LD foams before and after infiltration. In the images of the infiltrated samples, the white tinge on the sample surface is from the $MgCl_2$ PCM. Prior to the infiltration process, each graphite foam sample was weighed by means of a Mettler Toledo's scale with an accuracy of $\pm 0.01 \text{ g}$. After the infiltration process, the graphite foam samples infiltrated with $MgCl_2$ mass were then weighed by the same digital scale. The mass of solid $MgCl_2$ contained within the graphite foam corresponds to the increase in sample mass as follows:

$$m_{\text{MgCl}_2} = m_{\text{final sample}} - m_{\text{initial sample}} \quad (4)$$

The volume of solid MgCl_2 and liquid MgCl_2 was calculated from the solid and liquid density of MgCl_2 , as follows:

$$V_{\text{solid MgCl}_2} = \frac{m_{\text{MgCl}_2}}{\rho_{\text{solid MgCl}_2}} \quad (5)$$

$$V_{\text{liquid MgCl}_2} = \frac{m_{\text{MgCl}_2}}{\rho_{\text{liquid MgCl}_2}} \quad (6)$$

Then, the infiltration rate of the samples was calculated as follows [25]:

$$\text{Infiltration Rate} = \frac{V_{\text{solid MgCl}_2}}{\phi_{\text{foam}} V_{\text{foam}}} \quad (7)$$

$$\text{Infiltration Rate} = \frac{V_{\text{liquid MgCl}_2}}{\phi_{\text{foam}} V_{\text{foam}}} \quad (8)$$

where ϕ is the porosity of the graphite foam.

The infiltration rate of HD foam based on the solid volume of MgCl_2 was ~70%. However, the infiltration rate of HD foam based on the liquid volume of MgCl_2 was ~97%. This difference is because the volume of MgCl_2 increases as the phase of MgCl_2 changes from solid to liquid. Similarly, the infiltration rate of LD foam based on the liquid volume of MgCl_2 was ~98%, while the infiltration rate of LD foam based on the solid volume of MgCl_2 was ~71%. These results indicate that the graphite foams could almost be fully filled with MgCl_2 in the liquid phase.

Figure 4 shows SEM images of the MgCl_2 /HD foam composite. The pores are clearly filled with MgCl_2 . The boundary between the graphite foam and MgCl_2 can be seen, and the graphite ligament was not damaged by infiltration with PCM. The EDX results in Figure 4 confirm that the pores of the graphite foam were filled with MgCl_2 . As per EDX, there was presence of oxygen which is probably coming from any moisture absorbed in PCM from the environment. Similar observations were made for the LD foams.

(b) Thermal conductivity of foams with and without PCM

The measured thermal diffusivities of HD foam and MgCl_2 /HD foam composite in the z and x-y directions are plotted in Figure 5(a) as a function of temperature. For all samples, the thermal diffusivities decreased with increasing temperature. The thermal diffusivities of MgCl_2 /HD foam composites were lower than those of HD foam without MgCl_2 for both the z and x-y directions.

The thermal conductivities, as calculated from equation 1, of the HD foam and MgCl_2 /HD foam composites in the z and x-y directions are plotted in Figure 5(b) as a function of temperature. For all samples, the thermal conductivity decreased with increasing temperature. At room temperature, the calculated thermal conductivities of the MgCl_2 /HD foam composite and the HD

foam without MgCl_2 in the z direction were 269.8 ± 31.0 W/m-K and 232.5 ± 1.3 W/m-K, respectively. In the x-y directions the measured values of the composite and as-received HD foams were lower: 119.2 ± 6.7 and 92.8 ± 4.9 , respectively. For comparison, the manufacturer-provided thermal conductivities of the HD foam at room temperature in the z and x-y directions are 245 W/m-K and 70 W/m-K, respectively. The thermal conductivities of the MgCl_2 /HD foam composites were slightly higher than those of HD foam without MgCl_2 for the z and x-y directions. The infiltration of MgCl_2 into the graphite foam did not deteriorate the high bulk thermal conductivity of the foam.

At 600 °C, the calculated thermal conductivities of the MgCl_2 /HD foam composite and the HD foam without MgCl_2 in the z direction were 96.2 ± 13.8 W/m-K and 87.4 ± 1.2 W/m-K, respectively. The thermal conductivity of MgCl_2 /HD foam composites at 600 °C is ~200 times higher than that of pure MgCl_2 (0.46 W/m-K).

Similarly, for MgCl_2 /LD foam composites, the thermal diffusivities decreased with increasing temperature (Figure 6(a)). The thermal diffusivities of MgCl_2 /LD foam composites were lower than those of LD foam without MgCl_2 for both z and x-y directions because of infiltration of the MgCl_2 .

The thermal conductivities of the MgCl_2 /LD foam composites in the z and x-y directions are plotted in Figure 6(b) as a function of temperature. For all samples, the thermal conductivity decreased with increasing temperature. At room temperature, the measured thermal conductivities of the LD foam and MgCl_2 /LD foam composite in the z direction were 43.0 ± 0.3 W/m-K and 37.7 ± 5.4 W/m-K, respectively. The conductivities for the as-received foam and its composite were lower: 20.4 ± 0.3 W/m-K and 18.7 ± 1.7 W/m-K, respectively.

At 600 °C, the calculated thermal conductivities of the MgCl_2 /LD foam composite and the LD foam without MgCl_2 in the z direction were 14.6 ± 0.5 W/m-K and 16.7 ± 0.5 W/m-K, respectively. The thermal conductivity of the MgCl_2 /LD foam composite at 600 °C is about 30 times higher than that of pure MgCl_2 (0.46 W/m-K).

Unlike the HD foam case, the thermal conductivities of LD foam with MgCl_2 were slightly lower than that of the foam without MgCl_2 for the z and x-y directions. It should be noted that in the composite thermal conductivity estimation, the PCM was considered to be pure MgCl_2 with a conductivity value of 0.46 W/m-K. However, it is known that MgCl_2 is hygroscopic and absorbs water to form $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ on exposure to air. The density of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ at room temperature is 1.569 g/cm^3 , which is 1.5 times lower than that of MgCl_2 . Meanwhile, the specific heat capacity of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ at room temperature is 1.59 J/g-K , which is 2.1 times higher than that of MgCl_2 [32]. If the density and specific heat capacity of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (thermal conductivity value of 0.57 W/m-K) are used for the calculation of the thermal conductivity, the thermal conductivity of the LD foam/PCM composite in the z direction at room temperature is calculated to be 43.8 W/m-K , which is slightly higher than that of the foam without MgCl_2 in the z direction. This finding was observed only for the LD foam, probably because of the higher PCM content and, hence, larger propensity for moisture absorption.

Our investigation of the conductivity of the MgCl_2 /graphite foam composites suggests that the MgCl_2 /graphite foam composites could charge/discharge thermal energy rapidly. In other words, the limitation of the use of PCM as heat storage media for LHTES might be overcome by deploying graphite foam with very high thermal conductivity. Furthermore, the storing energy density and charging/discharging rate of LHTES can be controlled by the porosity of the graphite foam.

(c) TGA-DSC analysis of pure MgCl_2

The TGA-DSC analysis was conducted for both MgCl_2 /HD foam and MgCl_2 /LD foam composites to investigate the heat of fusion of the infiltrated foam. Prior to the TGA-DSC analysis of the infiltrated foams, pure MgCl_2 was evaluated. The measured melting temperature of the MgCl_2 was 710.3°C , which is about 0.5% lower than that of the literature value (714°C). The freezing temperature of the MgCl_2 was 700.4°C , and the corresponding super-cooling temperature of the MgCl_2 was 9.9°C . The measured heat of fusion of the MgCl_2 was 447.8 J/g , which is 1% lower than the literature value (452 J/g).

(d) TGA-DSC analysis of MgCl_2 /HD foam composite

Figure 7 shows the heat flow of the MgCl_2 /HD foam composite in the 1st cycle of melting and freezing, as well as the weight change of the MgCl_2 /HD foam composite for 100 cycles of melting and freezing. The heat of fusion of the MgCl_2 /HD foam composite was in the range from 225.2 kJ/kg to 238.5 kJ/kg for all cycles. The mass ratio of MgCl_2 in the MgCl_2 /HD foam composite was calculated by the measured heat of fusion of the MgCl_2 /HD foam composite. The mass ratio of MgCl_2 in the MgCl_2 /HD foam composite at the 1st cycle calculated by the measured heat of fusion was 49.8%, while the initial mass ratio of MgCl_2 was 53.7%. These results confirm that the calculation process (Eqs. (4)-(8)) for the infiltration rate was correct.

The curves of heat flow for 100 cycles are quite repeatable. This result indicated that the MgCl_2 /HD foam composite could continuously charge and discharge thermal energy. The weight of composites decreased rapidly for the early stage of melting and freezing cycles. Afterwards, the decrease of weight of composites slowed down. Initial rapid weight loss might be caused by vaporization of H_2O remaining inside of the composite, and the later weight loss might come from oxidation of graphite foam or vaporization of MgCl_2 . Note that the graphite foam could be coated by SiC to mitigate oxidation, and the atmosphere will be controlled with inert gas in the real LHTES system. Furthermore, the LHTES container will be sealed to avoid loss of MgCl_2 . Therefore, in a real LHTES system, there will be no weight loss of MgCl_2 /graphite foam composite. The measured melting temperature of MgCl_2 in the MgCl_2 /HD foam composite was in the range from 708.8°C to 709.9°C . The measured freezing temperature of MgCl_2 in the MgCl_2 /HD foam composite was in the range from 699.0°C to 700.5°C [33]. We concluded that the presence of HD foam did not affect the melting and freezing temperature of the MgCl_2 .

(e) TGA-DSC analysis of MgCl_2 /LD foam composite

Figure 8 shows the heat flow of the MgCl_2 /LD foam composite at the 1st cycle of melting and freezing, as well as the weight change of the LD foam infiltrated with MgCl_2 for 100 cycles of melting and freezing. The heat of fusion of LD foam infiltrated with MgCl_2 was in the range from 353.6 kJ/kg to 379.6 kJ/kg for all cycles. The mass ratio of MgCl_2 at the 1st cycle calculated

by using the heat of fusion is 84%, and the initial mass ratio of MgCl_2 is 86.7%. Again, this result confirms that the determination for the infiltration rate was consistent.

4. Conclusions

In the present study, two types of graphite foams with different porosities were infiltrated with MgCl_2 . After the infiltration of MgCl_2 /graphite foam composites, the heat of fusions, melting/freezing temperatures, and thermal diffusivities were measured and compared to those of pure MgCl_2 . The infiltration rates for both foam types were almost to full capacity based on the molten PCM. The TGA-DSC analyses of the MgCl_2 /graphite foam composites showed that the heat of fusion of the composite corresponded to the mass ratio of the MgCl_2 to the graphite foam. Also, it was shown that the melting/freezing temperatures of the MgCl_2 did not change in the presence of the graphite foams. From the conductivity data of the MgCl_2 /graphite foam composites, we concluded that the MgCl_2 /graphite foam composites could charge/discharge thermal energy rapidly. In other words, the limitation of the use of PCM as heat storage media for LHTES could be overcome by deploying a graphite foam with very high thermal conductivity. Furthermore, the storing energy density and charging/discharging rates of LHTES can be controlled by the porosity of the graphite foam.

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Table 1. Physical and Thermal properties of MgCl₂ [32]**Magnesium Chloride (MgCl₂)**

Density	Solid (at 25 °C) : 2.32 g/cm ³ Liquid (at 714 °C): 1.68 g/cm ³
Melting Temperature	714 °C
Heat of Fusion	452 J/g
Specific Heat Capacity	$0.7602 + 1.65671 \times 10^{-4} \cdot T$ (J/g-K); T is the absolute temperature
Thermal Conductivity	0.46 W/m-K (at 714 °C)
Under Cooling	~30 °C
Vapor Pressure	1 mm Hg at 778 °C 5 mm Hg at 877 °C 10 mm Hg at 930 °C

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Table 2. Sample dimensions and measurement conditions for thermal diffusivity

Sample Characteristics	
Diameter	12.7 mm
Thickness	3~7 mm
Material	<ul style="list-style-type: none">• HD foam in z direction• HD foam in x-y directions• MgCl₂/HD foam in z direction• MgCl₂/HD foam in x-y directions• LD foam in z direction• LD foam in x-y directions• MgCl₂/LD foam in z direction• MgCl₂/LD foam in x-y directions
Measurement Conditions	
Temperature	25 °C – 800 °C for graphite foam 25 °C – 600 °C for MgCl ₂ /graphite foam composite
Atmosphere	Nitrogen gas

Figure 1. Schematic of melt infiltration process.

(a)

(b)

Figure 2. SEM images of as-received graphite foams: (a) HD foam and (b) LD foam.

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Figure 3. HD and LD foams before and after infiltration.

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Label A: H2O_600C_5kV

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Figure 4. SEM images and EDX analysis of MgCl_2/HD foam composite.

Figure 5. (a) Thermal diffusivities and (b) thermal conductivities of HD foam with and without MgCl_2 in z and x-y directions.

Figure 6. (a) Thermal diffusivities and (b) thermal conductivities of LD foam with and without MgCl₂ in z and x-y directions.

Figure 7. TGA-DSC analysis for HD graphite foam infiltrated with MgCl_2 : (a) heat flow vs. temperature at 1st cycle and (b) weight change for 100 cycles.

Figure 8. TGA and DSC analysis for LD foam infiltrated with MgCl_2 : (a) heat flow vs. temperature at 1st cycle and (b) weight change for 100 cycles.