DOI: 10.15669/pnst.7.279

Progress in Nuclear Science and Technology
Volume 7 (2025) pp. 279-285

ARTICLE

High reactivity and durability of silicone-supported magnesium chloride composite material for ammonia using thermochemical energy storage

Tetta Enosawa [a], Saki Yoshida [a], Hiroki Takasu [b]* and Yukitaka Kato [b]

[a] *Graduate Major in Nuclear Engineering, Institute of Science Tokyo, 2-12-1-N1-22, Ookayama, Meguro-ku, Tokyo 152-8550;*
[b] *Laboratory for Zero-carbon Energy, Institute of Integrated Research, Institute of Science Tokyo,*
2-12-1-N1-22, Ookayama, Meguro-ku, Tokyo 152-8550

Thermochemical energy storage (TcES) is a promising candidate for innovative energy-storage technologies. In this study, magnesium chloride ($MgCl_2$)/ammonia (NH_3) TcES composite material, particularly suitable for effectively utilizing low-temperature waste heat of approximately 100 °C, were developed and evaluated using an NH_3-supplying thermobalance. A silicone-supported $MgCl_2$ composite material was prepared to suppress volume expansion caused by the progression of the NH_3 sorption reaction. The specific surface area of the prepared silicone-supported $MgCl_2$ material increased from 7.6 to 21.6 $m^2\ g^{-1}$ by the cyclic reaction, and the sorption number change achieved a theoretical value of 4.0. In addition, the heat-storage performance was observed to exhibit a heat storage density of 683 kJ L^{-1} and a maximum heat-storage rate of 0.668 kW L^{-1}. These results confirmed that silicone-supported $MgCl_2$ could suppress the volume expansion caused during the NH_3 sorption reaction while maintaining its high reactivity to NH_3. It was observed that silicone-supported $MgCl_2$ and NH_3 systems are promising material that can realistically be used for heat storage/output at approximately 100 °C.

Keywords: ***ammonia; magnesium chloride; thermochemical energy storage; silicone rubber; waste heat; composite material***

1. Introduction

Global concerns regarding global warming and climate change make it vital to utilize renewable energy sources [1]; however, the generating capacity of renewable energy resources fluctuates according to external factors. Therefore, there are situations when there is a surplus or shortage of electricity supply, and energy storage systems are required to ensure a stable supply. Among the energy storage systems, thermal energy storage materials have attracted attention recently owing to their low cost and long storage periods [2]. Thermal energy storage technologies include sensible heat storage, latent heat storage, and thermochemical energy storage (TcES).

TcES not only has a higher heat storage density than sensible and latent heat storage [2], but can also theoretically provide semi-permanent long-term storage of heat, as the reactant gas and heat storage material can be physically separated and stored. However, the practical application of TcES requires the development of materials for different target temperature ranges and reactions. The development of TcES materials for the target temperature range of approximately 100 °C, which accounts for a large proportion of waste heat [3], is important not only for energy leveling, but also for the effective utilization of waste heat.

Magnesium chloride is a promising TcES material in this temperature range, and has been selected owing to its high reactivity and low cost [4]. Systems using magnesium chloride and water have been investigated worldwide. However, the liquefaction phenomena of the material owing to the low melting point of the product and the difficulty of supplying sufficient reactive vapor pressure without an external energy supply in cold atmospheres, such as <0 °C, have been identified as problems [4]. To solve these problems, a TcES using NH_3 as the reaction gas has been proposed [5].

The $MgCl_2/NH_3$ system discussed in this study is expected to be used because the low boiling point of NH_3 allows it to operate in a cold atmosphere [6-8], and NH_3 is often handled in various chemical industries, including fertilizer plants. $MgCl_2/NH_3$ forms mono-, di-, and hexa-ammine complexes [9]. Applying the respective reaction enthalpies, ΔH_r, and reaction entropies, ΔS_r, identified in the literature [9] for each reaction to van 't Hoff's equation, the formation of hexa-ammine complexes from the di-ammine complex reaction can proceed reversibly at approximately 100–200°C without pressuring NH_3.

$$\mathrm{Mg(NH_3)_2Cl_2(s) + 4NH_3(g) \rightleftarrows Mg(NH_3)_6Cl_2(s)} \qquad (1)$$

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*Corresponding author. E-mail: takasu.h.aa@m.titech.ac.jp

Moreover, Takasu et al. reported that $Mg(NH_3)_2Cl_2$, obtained by the activation process, exhibits a high reactivity toward NH_3 and concluded that the $MgCl_2/NH_3$ pair is a promising candidate as a TcES material [10]. However, in the reactions in Eq. (1), when the NH_3 sorption reaction proceeds, $Mg(NH_3)_6Cl_2$ is formed with a large volume [11]. Mofidi et al. determined that the pure anhydrous $MgCl_2$ expands about 400% upon NH_3 sorption reaction [12]. This volume expansion causes various problems, such as a decrease in the heat storage density per volume and damage to the reactor, which is a major issue for TcES materials that must be solved to achieve practical application. To suppress the volume expansion and salt agglomeration caused by chemical reactions, Calabrese et al. developed a silicone-supported $SrBr_2/H_2O$ TcES material; however, its performance is unclear [13].

The objective of this study is to develop a silicone-supported $MgCl_2$ composite material, which is a composite of silicone and $MgCl_2$, in order to prevent volume expansion of $MgCl_2$ material due to reaction and consequent destruction of sample shape. In addition, repeated reactivity evaluation of the prepared silicone-supported $MgCl_2$ composite material as TcES was performed at 100°C to analyze the structural changes of the material before and after the reaction.

2. Experimental

2.1. Materials and sample preparation

Anhydrous magnesium chloride ($MgCl_2$, 98% purity, Kishida Chemical Corporation) was used as the initial sample. One-component room-temperature vulcanizing (RTV) silicone (KE-4895; Shin-Etsu Chemical Co., Ltd.) was used as the support. RTV silicone is a tough and durable elastomer that can be cured.

The samples were prepared according to the following procedure. RTV silicone and $MgCl_2$ anhydride were mixed in an Ar-substituted glove box at a mass ratio of RTV silicone:$MgCl_2$ = 4:6. The samples were then transferred to a desiccator and allowed to stand for 7 d. This allowed the polymerization of the silicone rubber to proceed. The obtained powder was mixed for 20 min using a mortar and pestle.

In order to maintain the shape of the compact during the reaction, $MgCl_2$ was reacted with NH_3 to convert it to the largest volume $Mg(NH_3)_6Cl_2$ state before the material was molded. The obtained powder was reacted with NH_3 for 4 h at 25 °C at an NH_3 flow rate, F_{NH3}, of 51.5 mL min^{-1} at standard ambient temperature and pressure, SATP, and then press-molded using a press apparatus (TB-20H, NPa Systems Ltd.) and a mold (inner diameter 7 mm) at 10 MPa for 3 min to produce the tablet shape. The tablets were adjusted to a height of approximately 4 mm and mass of approximately 120 mg. These composite materials were termed 'SR0.4 tablet' based on the mass ratio and the abbreviation of silicone rubber when mixed during the initial stages of preparation.

NH_3 sorption pretreatment was performed according to [10]. The composite materials were reacted with NH_3 for 6 h at 25 °C under an NH_3 supply pressure, P_{NH3}, of 100 kPa. The temperature was then increased to 100 °C without changing the atmosphere, held for 15 min, after which the heating was stopped. This procedure was termed 'reaction-activated tablet pretreatment'. XRD measurements were performed to confirm whether the reaction progressed sufficiently to the inside of the sample after ammonia sorption, and the chemical form of the heat storage material inside the material was confirmed to be $Mg(NH_3)_6Cl_2$ after NH_3 sorption. The following equation was used to calculate the mass ratio of $Mg(NH_3)_2Cl_2$ in the composite material:

$$a = \frac{m_{Mg(NH_3)_2Cl_2}}{m_{silicone} + m_{MgCl_2} \cdot \left(M_{Mg(NH_3)_2Cl_2} \middle/ M_{MgCl_2} \right)} \tag{2}$$

where a denotes the mass ratio of $Mg(NH_3)_2Cl_2$ in the composite material, M_X the molar mass of X, and m_X the mass of X. The mass ratio, a, was 0.76.

The volume ratio of $Mg(NH_3)_2Cl_2$ in the composite material, r_v, and the porosity of the composite material, ϕ, were calculated from the density of the composite material according to the Eqs. (3) and (4).

$$r_v = \frac{a \cdot \rho_{Mg(NH_3)_2Cl_2}}{(1-a) \cdot \rho_{silicone} + a \cdot \rho_{Mg(NH_3)_2Cl_2}} \tag{3}$$

$$\phi = 1 - \frac{\rho_{sample}}{(1-a) \cdot \rho_{silicone} + a \cdot \rho_{Mg(NH_3)_2Cl_2}} \tag{4}$$

where $\rho_{silicone}$, $\rho_{Mg(NH3)2Cl2}$, and $\rho_{silicone}$, are the are the densities of each material, which were 1.04, 1.70 [12], and 0.85 g cm^{-3}, respectively. The volume ratio, r_v, and porosity of the composite material, ϕ, were calculated as 0.79 and 0.29, respectively. The density of the composite material, $\rho_{silicone,}$ was calculated using the tablet diameter, height, and mass after the reaction-activated tablet pretreatment.

The NH_3 sorption/desorption cyclic experiments, to investigate the reactivity of the sample, were performed after the reaction activation tablet pretreatment and NH_3 'desorption pretreatment'. Desorption pretreatment was performed under a nitrogen (N_2) pressure, P_{N2}, of 100 kPa, and temperature of desorption pretreatment, T_{dp}, of 165°C while transforming the chemical form of the heat storage material inside the composite material to $Mg(NH_3)_2Cl_2$. After maintaining at 165°C for 1 h, the temperature was decreased to 100°C, and the desorption pretreatment was completed. Subsequently, a cyclic experiment was conducted immediately after desorption pretreatment under these conditions, and the sorption reaction was performed at a P_{NH3} of 100 kPa and sorption temperature, T_s, of 100 °C for 60 min. The desorption reaction was conducted at a P_{NH3} of 5 kPa and desorption temperature, T_d, of 120°C for 55 min.

As both the temperature and pressure conditions were changed for the sorption/desorption reactions, the temperature stabilization time was set at 10 min during the cyclic experiment.

2.2. Apparatus

An NH_3-supplying thermobalance apparatus was used for the experiments in an open system. The thermobalance was a TGD-9600 (ADVANCE RIKO, Inc.). A stream of N_2 and NH_3 gases was supplied above the sample chamber. NH_3 (purity > 99.999%, ShowaDenko K. K.) was used as the reactant gas. N_2 (purity > 99.999%, TAIYO NIPPON SANSO Corp.) was used as the carrier gas and purge gas to prevent contamination of the inside of the thermobalance by NH_3. The flow rate of each gas was controlled by a mass flow controller (MFC, MODEL 3660, KOFLOC Corp.), the total flow rate of the gas supplied to the sample chamber was fixed at 51.5 mL min^{-1} (SATP), and the NH_3 partial pressure was calculated from the flow rate of each gas. The details of the reaction evaluation apparatus have been described elsewhere [10].

Using the NH_3 supplying thermobalance apparatus, the mass change of the sample during the NH_3 sorption/desorption reaction was measured using a thermobalance to investigate the reactivity of the sample and NH_3.

The composite materials were observed using scanning electron microscopy (SEM; JCM-7000, JEOL Ltd.). All observations were performed at an acceleration voltage of 5 kV.

N_2 physisorption measurements were performed to determine the specific surface area and pore distribution. A high-precision specific surface area and pore distribution measuring instrument (BELSORP-max, MicrotracBEL Corp.) was used for these measurements.

2.3. Evaluation equation

The reactivity was evaluated using 'sorption number', based on the value of the mass change measured by the thermobalance apparatus. The sorption number is the amount of NH_3 mol sorbed or desorbed per 1 mol of $Mg(NH_3)_2Cl_2$. The evaluation equation was as follows:

$$n = \frac{(m - m_{\mathrm{ini}})/M_{\mathrm{NH_3}}}{m_{\mathrm{ini}} \cdot a / M_{\mathrm{Mg(NH_3)_2Cl_2}}} \qquad (5)$$

$$\Delta n_{i\text{-th,sorp/des}} = \left| n - n_{i\text{-th,sorp/des ini}} \right| \qquad (6)$$

where, n denotes the sorption number, m the mass at a given reaction time after the start of the cyclic experiment, m_{ini} the mass value when the cyclic experiment is initiated, and M_{x} the molar mass of X.

$\Delta n_{i\text{-th,sorp/des}}$ denotes the sorption number change in the sorption or desorption reactions. The subscript 'sorp' was used to indicate the sorption number change during the sorption reaction, and the subscript 'des' was used to indicate the sorption number change during the desorption reaction. $n_{i\text{-th,sorp/des ini}}$ denotes the initial value of the sorption number, n, when the sorption or desorption reaction started at the i-th cycle, where i indicates the cycle number ($0 \leq i \leq 10$).

A detailed description of $n_{i\text{-th,sorp/des ini}}$ is given below.

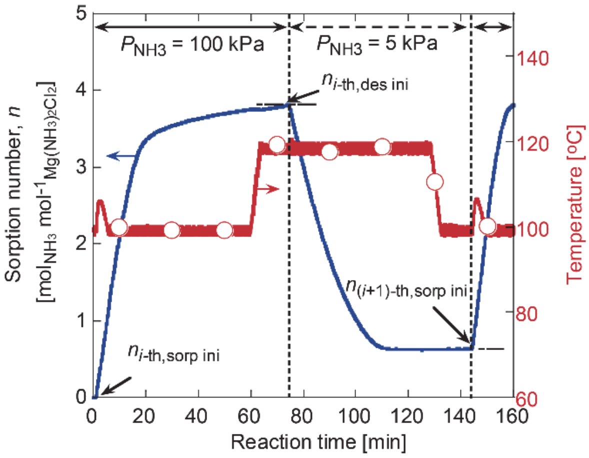

Figure 1. Definition of $n_{i\text{-th,sorp/des ini}}$ in each sorption/desorption reaction.

The following equation was used to evaluate the heat storage density per volume/mass.

$$q_{\mathrm{v,storage}} \text{ or } q_{\mathrm{m,storage}} = \frac{\left\{\left[m_{i\text{-th,des}}(t_{\mathrm{d}}) - m_{i\text{-th,des}}(0)\right]/M_{\mathrm{NH3}}\right\} \cdot \Delta H_{2-6}}{(V_{\mathrm{f}} \text{ or } m_{\mathrm{f}})} \qquad (7)$$

The following equation was used to investigate the heat storage rate per volume/mass of the composite material:

$$w_{\mathrm{v,storage}} \text{ or } w_{\mathrm{m,storage}} =$$

$$\frac{m_{i\text{-th,des}}(t_{\mathrm{d}}) - m_{i\text{-th,des}}(t_{\mathrm{d}}')}{t_{\mathrm{d}} - t_{\mathrm{d}}'} \cdot \frac{\Delta H_{2-6}}{M_{\mathrm{NH_3}} \cdot (V_{\mathrm{f}} \text{ or } m_{\mathrm{f}})} \qquad (8)$$

where $q_{\mathrm{v,storage}}$ and $q_{\mathrm{m,storage}}$ denote the heat storage density per volume and mass, respectively, and $w_{\mathrm{v,storage}}$ and $w_{\mathrm{m,storage}}$ denote the heat storage rate per volume and mass, respectively. $\Delta H_{2\text{-}6}$ denotes the reaction enthalpy in Eq. (1), ΔH_{r} = 55.7 kJ $mol^{-1}{}_{\mathrm{NH3}}$ [9]. $m_{i\text{-th,des}}(t_{\mathrm{d}})$ denotes the mass value at a given desorption reaction time, t_{d}, in the desorption reaction of the i-th cycle. $m_{i\text{-th,des}}(0)$ denotes the mass value at $t_{\mathrm{d}} = 0$, and the mass value at the time of the desorption reaction was initiated. V_{f} denotes the final volume of the composite material and m_{f} the final mass value of the composite material. We defined $t_{\mathrm{d}} - t_{\mathrm{d}}' = 60$ s.

For the calculation of q_{storage} and w_{storage} in Eqs. (7) and (8), when the subscript is v (referring to volume), the volume value is used as the denominator, and when the subscript is m (referring to mass), the mass value is used as the denominator.

3. Results and discussions

3.1. Reactivity and durability evaluation from cyclic reaction

From the experimental results of the mass change associated with the NH_3 sorption and desorption reactions, we calculated the actual sorption number change in each sorption or desorption reaction using Eqs. (5) and (6). The behaviors of $\Delta n_{i\text{-th,sorp}}$ and $\Delta n_{i\text{-th,des}}$ in each cycle are shown below.

A plateau indicating the end of the sorption reaction was observed after approximately 20 and 30 min, respectively, for the desorption reaction in each cycle. Only the 1st cycle of the sorption reaction exhibited a peculiar behavior, and around the 2nd and 3rd cycles, both the sorption and desorption sides remained at approximately $\Delta n_{i\text{-th,sorp/des}} = 3.3$ to 3.4, but the final conversion increased with each cycle and stabilized around the theoretical value of 4 from the 8th cycle onwards. The initial sorption behavior may be attributed to the partial formation of mono-ammine complexes owing to the pretreatment at 165°C.

These experimental results confirm the completion of the reaction within 30 min. The high reactivity of $MgCl_2$ and NH_3 was not lost when silicone rubber was used as the supporting material. Silicone was observed to act as an excellent supporting material that did not inhibit the $MgCl_2$ and NH_3 reactions. The powder $Mg(NH_3)_2Cl_2$ reported earlier [10] was also tested under same sorption conditions (100°C, under 100 kPa of NH_3), and the reaction was completed in about 7 minutes. Although it is difficult to make a simple comparison due to the different amount of samples used, the sorption time of the composite material took longer than that of the powder material. This may be due to a slight decrease in NH_3 diffusivity in the composite material due to the use of silicone as a support, or to an increase in the local temperature of the material due to the heat generated during the sorption reaction, which may have decreased the reaction rate.

The appearance of the composite materials before and after the cyclic reaction is shown in **Figure 3**. Notably, the chemical form of the $MgCl_2$ complex inside the composite material before the cyclic experiment was $Mg(NH_3)_6Cl_2$, which was obtained after the end of the reaction activation-tablet pretreatment, whereas the chemical form of the heat storage material inside the composite material after the cyclic experiment was $Mg(NH_3)_2Cl_2$ after the desorption reaction in the last cycle.

The material volume obtained from the dimensions assuming the material as a cylindrical shape was 172 mm^3 in Figure 3 (a), whereas it was 176 mm^3 in Figure 3 (b), a change of only about 2%. This result confirms that the use of silicone material as a support can suppress shape fracture due to the volume change associated with the reaction of $MgCl_2$ with NH_3. This effect may also have been caused by the elasticity of the silicone rubber. Moreover, silicone rubber was shown to be effective in maintaining the tablet shape and reducing tablet cracking.

These results confirm that silicone-supported $MgCl_2$ can macroscopically maintain its original shape after 10 cycles, while maintaining the high reactivity of the $MgCl_2/NH_3$ system.

Subsequently, SEM observations of the top surface of composite materials after the cyclic experiments were performed to confirm that the microstructure was maintained. The results of SEM observations of the apex are shown below.

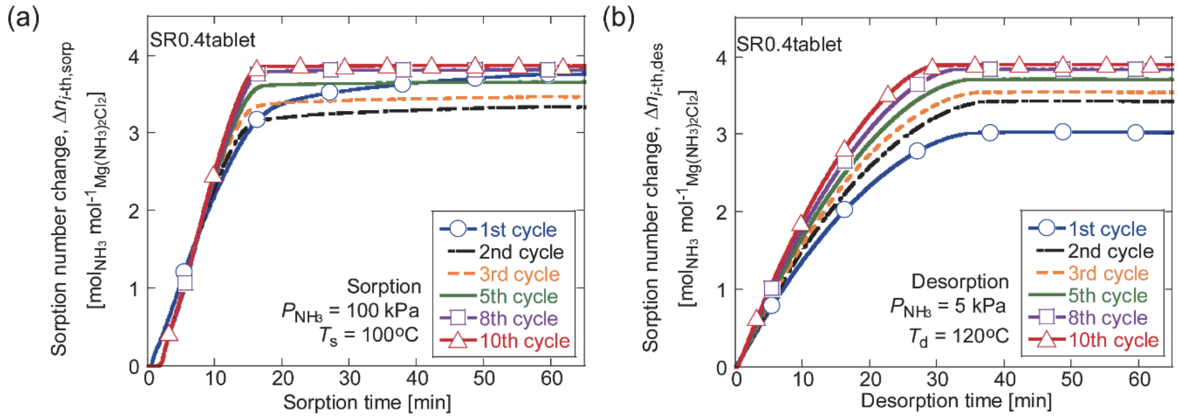

Figure 2. Result of the $\Delta n_{i\text{-th,sorp/des}}$ behavior in cyclic experiment. (a) Sorption reaction in each cycle, and (b) desorption reaction in each cycle.

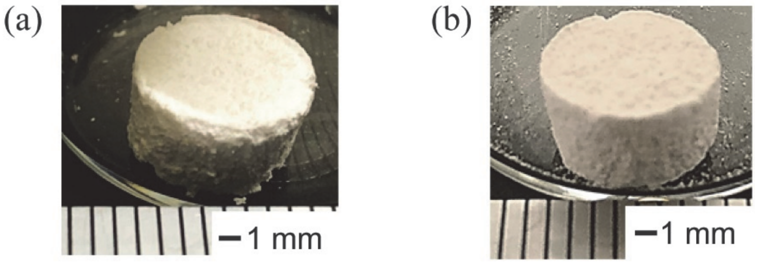

Figure 3. Change of appearance of SR0.4 tablet before and after 10 cycles. (a) Before cyclic experiment (after reaction activation-tablet pretreatment), and (b) after 10 cycles.

SEM observations before and after the cyclic experiments confirmed that the interconnected structure was maintained, with no exfoliation of $MgCl_2$ and silicone in the µm order. The silicone rubbers were well connected by engulfing crystalline $MgCl_2$, which may have contributed to the improved flexibility of the composite material. Simultaneously, a trend toward an increase in $MgCl_2$ particle size was observed. The voids observed by SEM suggested that the voids were employed as a gas diffusion pathway for NH_3, whereas the interconnected structures helped the silicone rubber elasticity to operate correctly. This confirms that the microstructure is maintained.

N_2 physisorption measurements were conducted to investigate the pore size and specific surface area. The results of the N_2 adsorption experiment and the pore size distribution calculated using the Barrett-Joyner-Halenda (BJH) method before and after the cyclic experiments are described below. $a_{s,BET}$ denotes the specific surface area calculated by the Brunauer-Emmett-Teller (BET) method, V_a the N_2 adsorption amount per gram of composite material at the standard temperature and pressure , STP, V_p the pore volume, and r_p the pore radius.

From these results, a threefold increase in the specific surface area, from 7.01 to 21.6 m^2 g^{-1}, was observed before and after the cyclic experiment. In addition, peaks at approximately 3 and 30 nm in the pore size distribution were only observed before the cyclic reactions, but after the cyclic experiment, a peak was detected at approximately 12.2 nm. These results suggest that the cyclic experiments increase the specific surface area and promote pore formation. It is suggested that this increase in the specific surface area and the progression of the pore formation influence the behavior of the final sorption number. In a previous report [10], it was confirmed that NH_3 sorption under room temperature as a pretreatment for reaction activation of $MgCl_2$, followed by NH_3 desorption, produced pores of about 5 nm. In addition, the specific surface area of the material in that case was 16.9 m^2 g^{-1}, which is a larger value than the specific surface area of the material reported here after repeated experiments. Considering these results, it seems possible that NH_3 sorption/desorption formed pores of about 5 nm as a diffusion pathway for NH_3, and that this pore size subsequently grew with repeated reactions.

Table 1. Specific surface area of SR0.4 tablet calculated from N_2 physisorption result.

status	$a_{s,bet}$ [m^2 g^{-1}]
before cyclic experiment	7.01
after cyclic experiment	21.6

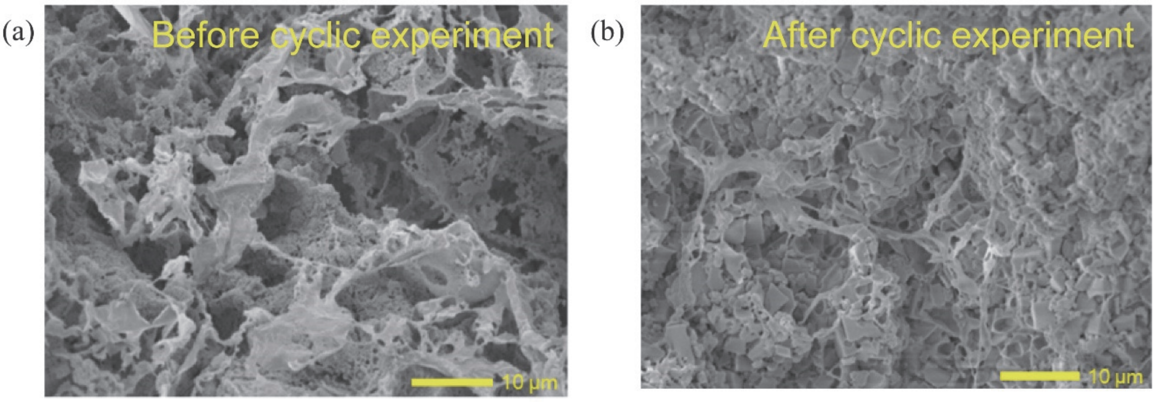

Figure 4. SEM images of top surface of SR0.4 tablet before and after 10-cycle experiment. The acceleration voltage is 5 kV and the magnification is 2000×. (a) Top surface before the experiment, and (b) top surface after the experiment.

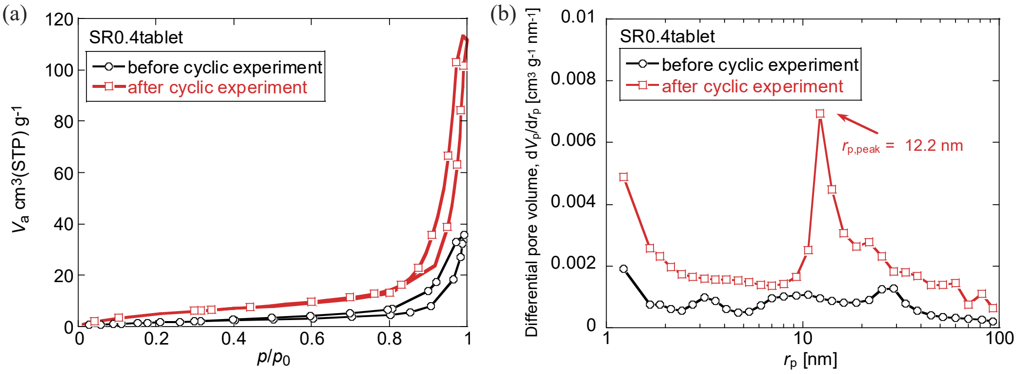

Figure 5. Results of N_2 physisorption measurement of SR0.4 tablet before and after 10-cycle experiment. (a) Isotherm and surface area calculated by the BET method, and (b) pore distribution calculated by the BJH method.

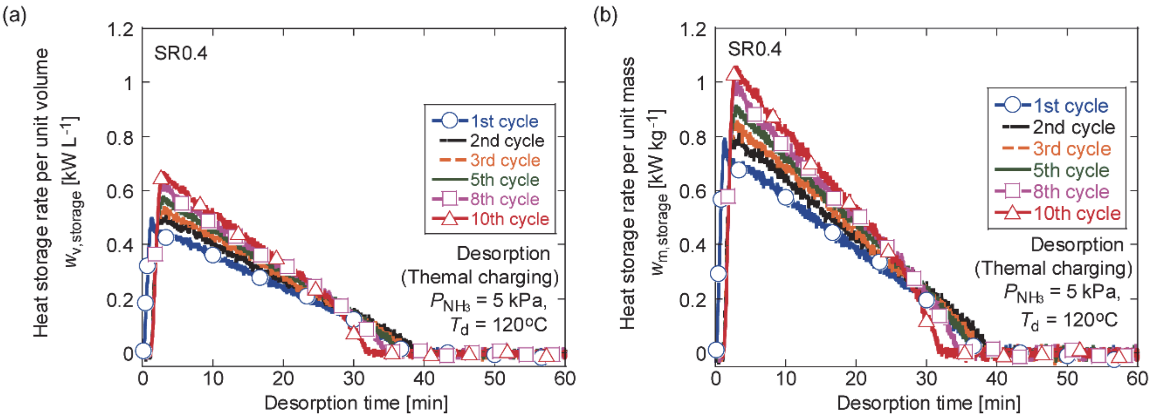

Figure 6. Heat storage rate per unit volume or mass. (a) Storage rate per unit volume of SR0.4 tablet in each cycle, and (b) storage rate per unit mass of SR0.4 tablet in each cycle.

3.2. Thermal evaluation from cyclic experiment

In this section, the heat storage performance of the material is evaluated based on the heat storage density and rate of the composite material created in this study. The volume of the material was calculated based on the diameter and height of the tablets. The heat storage rate per mass/volume for each cycle is expressed as follows:

At each rate, the maximum value was obtained within 10 min. The maximum heat-storage rate increased as the number of cycles increased. This indicates that the heat-storage performance did not deteriorate during the 10-cycle experiment, confirming that the created composite material possessed a rapid heat storage ability.

The maximum heat storage density per mass and volume was determined as 683 kJ L^{-1} and 1.08 MJ kg^{-1}, respectively, and the maximum heat storage rate per mass and volume was determined as 1.06 and 0.668 kW L^{-1}, respectively.

From these results, it can be emphasized that this silicone-supported $MgCl_2$ composite material is a promising TcES material at approximately 100 °C.

4. Conclusions

In this study, a $MgCl_2/NH_3$-based TcES material was developed to utilize low-temperature waste heat and energy storage effectively. The silicone-supported $MgCl_2$ composite material reached a theoretical sorption number change of 4 in the 10th cycle for both sorption and desorption reactions. Moreover, its volume remained unchanged in the millimeter-scale after the cyclic experiment. SEM images indicated that the microstructure was maintained. It was confirmed that the material created in this study can suppress volume expansion owing to its elasticity. A maximum heat storage performance per volume of 683 kJ L^{-1} and heat storage rate per volume of 0.668 kW L^{-1} were achieved, indicating that it is an excellent heat storage material.

These results indicate that the silicone-supported $MgCl_2$ composite material proposed in this study is an excellent material for low-temperature waste heat/energy storage with high reactivity to NH_3 and a decreased volume expansion.

Acknowledgements

This work was supported by JSPS KAKENHI Grant Numbers 19K15488. We would like to express our deepest gratitude for this support.

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