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Experimental Study on Synergistic Enhancement of Thermophysical Properties of Ternary Carbonates by Multidimensional Nanoparticles

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ABSTRACT

Molten salts have the advantages of a wide range of liquid temperatures and high heat storage capacity, which have been widely used in the field of solar thermal utilization. The significant disadvantage of molten salts is their low thermal conductivity, and the addition of nanoparticles can effectively enhance the heat transfer ability of molten salts. In this paper, novel composite molten salt materials are prepared by adding zero-dimensional Al_2O_3 nanoparticles, one-dimensional multi-walled carbon nanotubes, and two-dimensional graphene nanosheets with different combinations of multidimensional nanoparticles, respectively, using ternary carbonates as the base salt. The thermal diffusivity of the composite carbonates in the liquid state was measured by the laser flash method at different temperatures to analyze the effect of multidimensional nanoparticles on the thermophysical properties of ternary carbonates. The experimental results show that zero-dimensional alumina nanoparticles and two-dimensional graphene sheets have a synergistic strengthening effect. With the addition of zero-dimensional alumina nanoparticles and an additional 0.5% mass fraction of two-dimensional graphene nanosheets, the thermal diffusivity of the composite carbonate can be enhanced by a maximum of 54.08%, and the prepared composite carbonate has a better stability.

Keywords: nanoparticles, molten salts, heat transfer enhancement, thermal diffusivity

NONMENCLATURE

Symbols	
c_p	Specific heat capacity
d	Thickness of sample
λ	Thermal conductivity
ρ	Density
α	Thermal diffusivity
$t_{1/2}$	Time to reach half maximum temperature

1. INTRODUCTION

With the development of human society, the energy demand is growing. The problem of environmental pollution brought about by the use of traditional fossil energy sources has also become more serious, so clean energy sources, such as nuclear and solar energy, have become increasingly important. Molten salt thermal storage is a technology that utilizes the high specific heat capacity and high latent heat of phase change of molten salt to store and release thermal energy. It has the advantages of high heat storage density, a wide range of heat storage temperatures, high heat storage efficiency, and environmental friendliness [1]. It can be applied to heat storage of various heat sources. As an ideal heat storage medium, the development of molten salt heat storage technology has received wide attention over the world and has been applied in the fields of solar tower power generation, solar trough power generation, solar Fresnel power generation, etc. It also shows great potential in the fields of nuclear energy and industrial waste heat recovery [2-4].

However, the disadvantages of molten salt such as low thermal conductivity also tend to cause problems such as oversized heat transfer/storage systems and low heat transfer/storage efficiency. Therefore, in recent years, there has been a gradual increase in the research on the modification of the thermophysical properties of molten salts, especially the addition of various types of

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nanoparticles to molten salts to form composite molten salt materials to improve their thermal conductivity [5-8]. Nanoparticles can be categorized according to their dimensionality as zero-dimensional (limited by the nanoscale in all three directions), one-dimensional (limited by the nanoscale in only two directions), and two-dimensional (limited by the nanoscale in only one direction). Wei et al. [9] measured the thermal conductivity of solar salts ($NaNO_3$:KNO_3 = 60:40, wt.%) with the addition of zero-dimensional MgO nanoparticles using the laser flash method, and the experimental results showed that the maximum enhancement of the thermal conductivity of the composite molten salt could reach up to 62.1%. Myers et al. [10] added different mass fractions of zero-dimensional CuO nanoparticles to $NaNO_3$:KNO_3 eutectic salt, and their results showed that the thermal conductivity enhancement of the composite molten salt could be up to 50%. Dokutovich et al. [11] added zero-dimensional α-Al_2O_3 nanoparticles to ternary carbonates (Li_2CO_3: Na_2CO_3: K_2CO_3 = 43.5%: 31.5%: 25.0%, mol.%), and the experimental results showed that the thermal conductivity of the composite molten salts increased with the increase in the volume fraction of α-Al2O3 nanoparticles added.

Although the addition of zero- or one-dimensional nanoparticles to molten salts is effective in enhancing their thermal conductivity, a weakening effect also occurs when the concentration of the addition is too high. Awad et al. [12] respectively added zero-dimensional Fe_2O_3 and CuO nanoparticles to the solar salt, and the experimental results showed that the composite molten salt with the addition of the two zero-dimensional nanoparticles, respectively, showed an increase and then a decrease in thermal conductivity and that the decrease in thermal conductivity was caused by nanoparticle agglomeration or precipitation. Madathil et al. [13] measured the thermal conductivity of ternary nitrate-zero-dimensional CuO nanoparticles composite molten salt by using the transient planar heat source method. The measurement results showed that the thermal conductivity of the composite molten salt increased and then decreased with the mass fraction of added CuO nanoparticles. The optimum addition ratio is 0.5%, and the decrease in thermal conductivity may be due to the inhomogeneous dispersion of nanoparticles. Yuan et al. [14] added different mass fractions of one-dimensional multi-walled carbon nanotubes to binary carbonates, and the experimental results showed that the thermal conductivity of the composite molten salts also increased and then decreased with the increase of the mass fraction of multi-walled carbon nanotubes, and reached a maximum value of 1.04 W/(m-K) with the addition of 0.75% of multi-walled carbon nanotubes, at which time the enhancement of thermal conductivity was 50.72%. As for low-temperature materials, it has been found that two-dimensional nanoparticles can form a good heat transfer network with zero-dimensional nanoparticles to improve the thermal conductivity of composites [15-18], but few studies have been reported in the field of high-temperature molten salts.

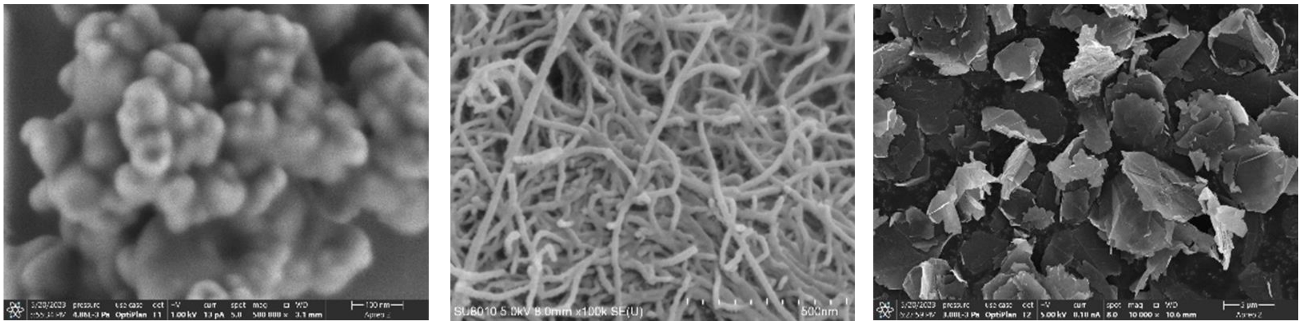
In this paper, ternary carbonates (Li_2CO_3: Na_2CO_3: K_2CO_3 = 40%: 30%: 30%, wt.%) were selected as the base salts to prepare zero-dimensional and multi-dimensional composite carbonate materials, respectively. The strengthening effect of multi-dimensional nanoparticles on the thermophysical properties of ternary carbonates was investigated by adding nanoparticles of different dimensions. The advantages of large specific surface area and easy dispersion of two-dimensional nanoparticles are utilized to study the synergistic enhancement of the thermophysical properties of two- and zero-dimensional nanoparticles on the thermal diffusivity of carbonates.

2. MATERIAL AND METHODS

2.1 Materials

The molten salt used in the experiments was Li_2CO_3-Na_2CO_3-K_2CO_3 (40-30-30, wt.%), and the highly thermally conductive reinforcing nanoparticles used were zero-dimensional alumina nanoparticles, one-dimensional multi-walled carbon nanotubes, and two-dimensional graphene nanosheets, respectively. The microstructures of the three nanoparticles were observed using Scanning Electron Microscope (SEM), and the results are shown in Fig. 1. It can be found that the average particle size of alumina nanoparticles is 30 nm. The diameter of multi-walled carbon nanotubes is 5-15 nm, and the length is 10-30 μm. However, both kinds of particles show obvious agglomeration phenomena under normal conditions, especially multi-walled carbon nanotubes, which are prone to entangled nodules. The graphene nanosheets were well dispersed, with a thickness of 4-20 nm and an average diameter of 5-10 μm. Because of the agglomeration of nanoparticles, ultrasonic shaking was adopted in the experiment to achieve uniform dispersion of nanoparticles in the base salt.

For the experiment, the nanoparticle composite molten salt was prepared by the aqueous solvation method in a two-step process, and the preparation flow is shown in Fig. 2. First, the three carbonates were



(a) Al_2O_3 nanoparticles (b) multi-walled carbon nanotubes (c) graphene nanosheets

Fig. 1. Scanning electron microscopy image of three nanoparticles

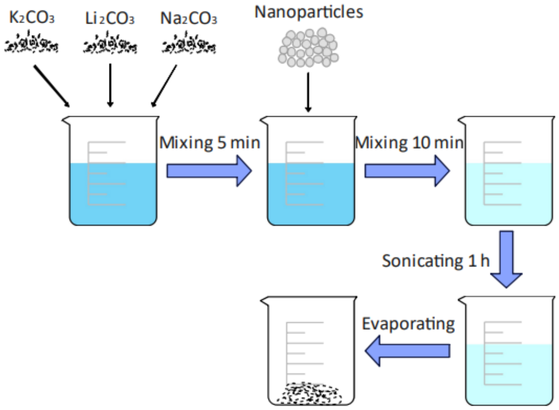


Fig. 2. Flow of ternary carbonate composites prepared by aqueous solution method

proportionally weighed (Li_2CO_3-Na_2CO_3-K_2CO_3 = 40-30-30, wt.%) and placed in a beaker, followed by the addition of deionized water and full dissolution of the ternary carbonates to obtain the aqueous ternary carbonate solution.

Next, zero-dimensional alumina nanoparticles, one-dimensional multi-walled carbon nanotubes, and two-dimensional graphene nanosheets were proportionally (0.5%, 1%, 1.5%, and 2%, wt.%) placed in the aqueous ternary carbonate solution, respectively. The mixed solutions were placed in an ultrasonic shaker (CD-4860, Codyson Company) for 1 hour to fully disperse the nanoparticles. Single nanoparticle composite carbonate materials were produced after stirring, evaporation, and drying processes the mixed solutions.

Next, nanoparticles of different dimensions with a mass fraction of 1% and a ratio of 1:1 were respectively placed in the aqueous carbonate solution and processed by stirring, evaporation, and drying to produce a multi-dimensional nanoparticle composite carbonate material.

Finally, zero-dimensional alumina nanoparticles with different mass fractions (0.5%, 1%, 1.5%, and 2%) and 0.5% two-dimensional graphene nanosheets were placed together in the aqueous carbonate solution and then processed by stirring, evaporation, and drying to produce a composite carbonate material with synergistic two-dimensional and zero-dimensional nanoparticles.

2.2 *Methods*

Commonly used thermal conductivity test methods can be divided into the steady state method and the transient method. In this experiment, we used the laser flash method to measure thermal conductivity. The flash method, which is one of the transient method, was first proposed by Parker [19]. Compared to other thermal conductivity test methods, the laser flash method has the advantages of shorter measurement time, less sample required and weak convective heat transfer caused by inhomogeneous temperature sites generated in the sample [20]. In the experiment, under a certain set temperature, a pulsed laser is emitted by the laser source in an instant and uniformly irradiated on the lower surface of the sample, so that the temperature of the surface rises instantaneously after absorbing the light energy, and the corresponding temperature rise process in the center part of the upper surface is continuously measured by an infrared detector. The temperature vs. time curve is obtained, and the thermal diffusivity is calculated according to Parker's formula:

$$\alpha = 0.1388\frac{d^2}{t_{1/2}} \tag{1}$$

Where α, d, and $t_{1/2}$ are the thermal diffusivity of the sample, the thickness of the sample, and the time required for the temperature of the upper surface of the sample to rise to half of its maximum value after receiving the laser pulse, the measured parameters can be used to calculate the thermal diffusivity of the sample by using equation (1). After obtaining the thermal diffusivity, use the conversion formula for thermal conductivity to thermal diffusivity:

$$\lambda = \alpha \cdot c_p \cdot \rho \tag{2}$$

Where λ, c_p, and ρ are the thermal conductivity, specific heat capacity, and density of the sample, respectively, which are calculated to obtain the thermal conductivity of the measured sample.

The laser flash method equipment used in this paper is the LFA457 Laser Flash Thermal Conductivity Meter, manufactured by NETZSCH. The samples were prepared by melting the salt at approximately 20 K above its melting point in platinum rhodium alloy crucible. And measurements were run under argon atmosphere at a flow rate of approximately 100 mL/min, the heating rate is 20 K/min, each interval 15 K read three flash points until 753.15 K.

This paper measured the thermal diffusivity of a standard specimen, Inconel-600, using the LFA457 Laser Flash Thermal Conductivity Meter to determine the accuracy of the instrument. The measured thermal diffusivity of Inconel-600 is shown in Fig. 3, and the diameter of Inconel-600 selected for the test was 12.58 mm with a thickness of 2 mm. Test results show that the thermal diffusivity of Inconel-600 deviates less than 5% from the standard value [21].

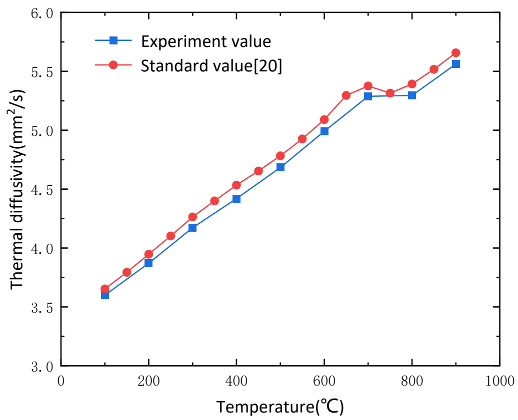


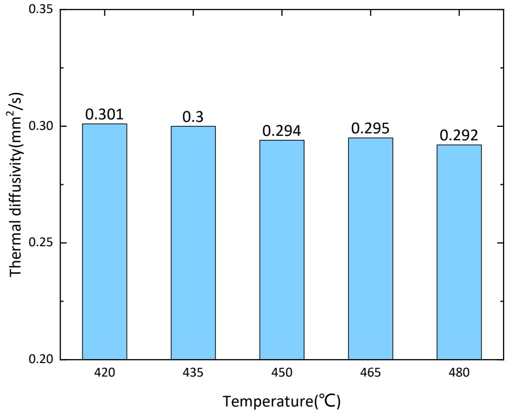
Fig. 3. Thermal diffusivity of Inconel-600 at different temperatures

3. RESULTS AND DISCUSSION

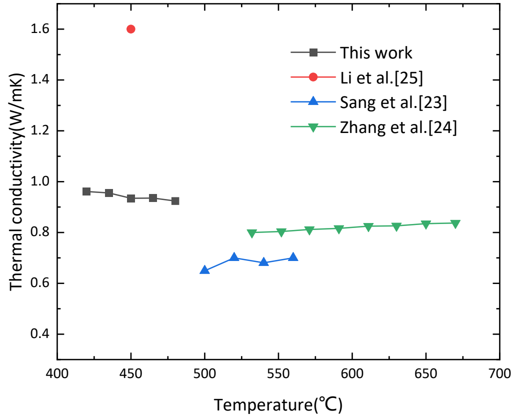
3.1 Thermophysical characterization of composite carbonates with zero-dimensional nanoparticles

3.1.1 Thermal diffusivity and thermal conductivity of ternary carbonates

The thermal diffusivity of the ternary carbonates was measured using a laser flash thermal conductivity meter, and the test results are shown in Fig. 4(a). The test results show that the thermal diffusivity of the experimentally configured ternary carbonate material decreases with increasing temperature. The specific heat capacity C_p and density ρ of ternary carbonates can be calculated from the fitted equations (3) and (4) in the literature [22], respectively. Finally, the carbonate thermal conductivity was calculated from equation (2) and compared with literature values [23-25] as shown in Fig. 4(b), where the experimentally measured data are within the literature measurement range.



(a) Thermal diffusivity of ternary carbonates



(b) Thermal conductivity of ternary carbonates

Fig. 4. Thermal diffusivity and thermal conductivity of ternary carbonates at different temperatures

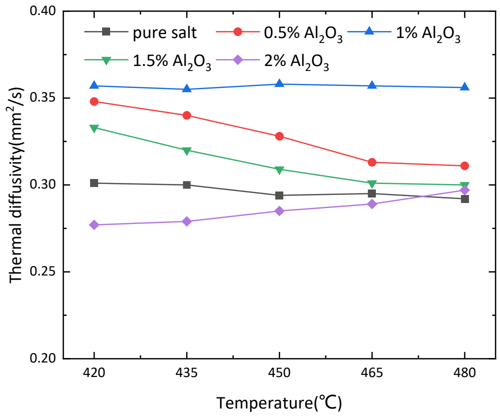
$$C_p = 1.54 + 0.116 \times 10^{-3} T \tag{3}$$

$$\rho = 2.27 - 0.434 \times 10^{-3} T \tag{4}$$

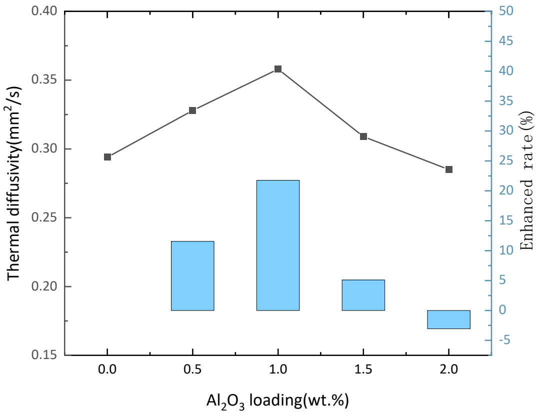
3.1.2 Strengthening effect of zero-dimensional Al_2O_3 nanoparticles

The thermal diffusivities of zero-dimensional Al_2O_3 nanoparticle composite carbonate materials with added mass fractions of 0.5%, 1%, 1.5%, and 2% were measured, and the thermal diffusivities obtained from the tests are shown in Fig. 5(a). The test results showed that the thermal diffusivity of the composite carbonate was maximized with the addition of 1% Al_2O_3 nanoparticles, and the maximum thermal diffusivity enhancement was 21.92%. The samples with the addition of 0.5% and 1.5% nanoparticles were not stable, and the measured thermal diffusivity fluctuated greatly with increasing temperature. The samples with 2% Al_2O_3 nanoparticles added even showed a weakening of the thermal diffusivity. As shown in Fig. 5(b), the thermal diffusivities of the composite carbonates were enhanced by 11.56%, 21.77%, 5.10%, and -3.06% at 450 °C with the addition of 0.5%, 1%, 1.5%, and 2% of Al_2O_3 nanoparticles, respectively. When the mass fraction of nanoparticles is small, the thermal conductivity of the composite molten salt is significantly enhanced due to the Brownian motion of the nanoparticles themselves and the compressive boundary layer formed on the surface of the particles [26]. When the mass fraction of nanoparticles was too large, the agglomerates formed by the nanoparticles were prone to settling, which hindered the further transfer of heat, thus leading to a decrease in the thermal conductivity of the composite molten salt.

Analyze the types of elements contained in the sample and their distribution using an Energy Dispersive Spectrometer (EDS). Fig. 6 shows the energy spectrum of the composite carbonate with 1% Al_2O_3 nanoparticles added, which can be seen from the distribution of Al element, which is more uniformly distributed in the composite material, indicating that the samples prepared by the aqueous solution method have good dispersion. In addition, comparing the EDS distribution of the Al elements with SEM images shows that the morphology corresponds well with the elemental

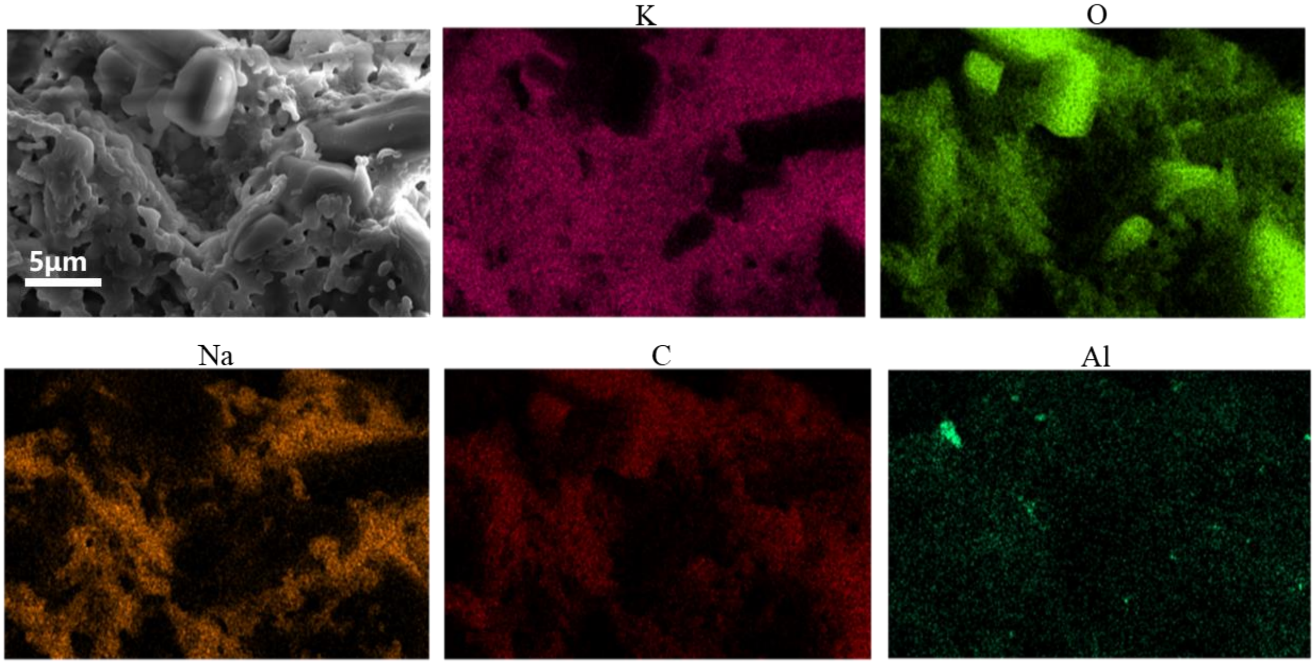


(a) Thermal diffusivity



(b) Thermal diffusivity enhancement

Fig. 5. Thermal diffusivity and thermal diffusivity enhancement of composite carbonates with different mass fractions of Al_2O_3 nanoparticles added at different temperatures



(a) Al_2O_3 nanoparticles (b) multi-walled carbon nanotubes (c) graphene nanosheets

Fig. 6. EDS image of composite carbonates with 1% Al_2O_3 nanoparticles added

distribution, which proves the accuracy of the EDS analysis.

3.2 Thermophysical characterization of composite carbonates with multi-dimensional nanoparticles

3.2.1 Synergistic thermophysical enhancement of multi-dimensional nanoparticles

Nanoparticles of different dimensions were added to ternary carbonates to investigate the effect of multi-dimensional nanoparticles on the thermal diffusivity of molten salts. Three composite carbonate materials with different dimensional nanoparticles added were prepared by selecting a 1% mass fraction in a 1:1 ratio, and their thermal diffusivity test results are shown in Fig.7. The test results showed that the maximum thermal diffusivity enhancement of the sample with zero-dimensional Al_2O_3 nanoparticles and two-dimensional graphene nanosheets was 42.85%, which was slightly lower than that of the composite carbonate sample with only graphene nanosheets. In contrast, the thermal diffusivity of the sample with the addition of Al_2O_3 nanoparticles and multi-walled carbon nanotubes, as well as the sample with the addition of multi-walled carbon nanotubes and graphene nanosheets, did not have an enhancing effect.

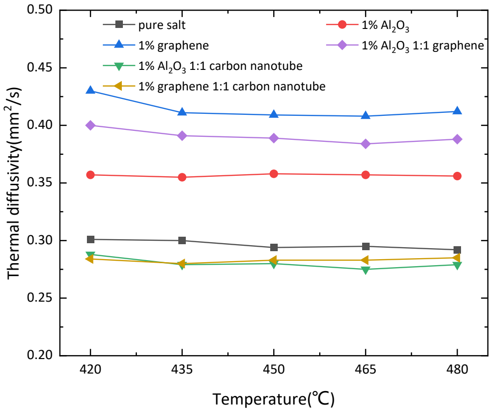
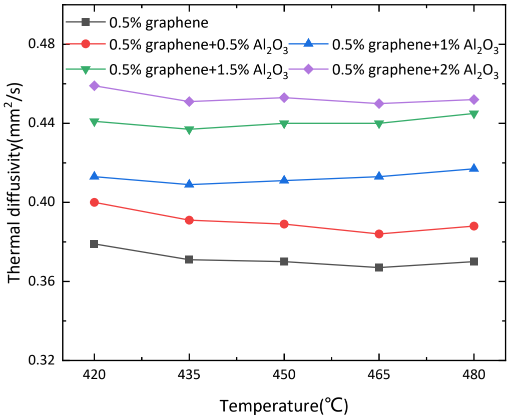


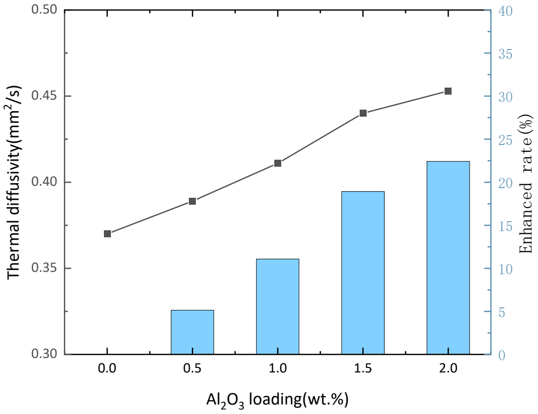
Fig. 7. Thermal diffusivity of composite carbonates with 1% addition of nanoparticles of different dimensions at different temperatures

By observing the scanning electron microscope images of one-dimensional multi-walled carbon nanotubes, it can be seen that the multi-walled carbon nanotube materials used in the experiments have undergone the phenomenon of entanglement and agglomeration, which is difficult to disperse by using an ultrasonic oscillator and results in the heat accumulating in the agglomerates and being difficult to be further transmitted. Therefore, the synergistic effect of one-dimensional multi-walled carbon nanotube materials with zero-dimensional Al_2O_3 nanoparticles and two-dimensional graphene nanosheets in the experiments was poor, resulting in the thermal diffusivity of the Al_2O_3 nanoparticles and multi-walled carbon nanotubes, as well as the samples with the addition of multi-walled carbon nanotubes and graphene nanosheets, not being enhanced.

3.2.2 Synergistic thermophysical enhancement of zero-dimensional and two-dimensional nanoparticles



(a) Thermal diffusivity



(b) Thermal diffusivity enhancement

Fig. 8. Thermal diffusivity and enhancement of adding 0.5% graphene with different mass fraction of Al_2O_3 particles at different temperatures

The degree of settling of zero-dimensional nanoparticles can be attenuated by taking advantage of the large specific surface area and easy dispersion of two-dimensional nanomaterials. Experiments were conducted to investigate the synergistic thermo-physical property enhancement of zero-dimensional and two-dimensional nanoparticles by adding zero-dimensional alumina nanoparticles with different mass fractions (0.5%, 1%, 1.5%, and 2%) with an additional addition of two-dimensional graphene nanosheets at a mass fraction of 0.5% to investigate the synergistic thermo-physical property enhancement of zero-dimensional and two-dimensional nanoparticles. The test results are shown in Fig. 8 (a). The test results show that the addition of different mass fractions of Al_2O_3 nanoparticles can enhance the thermal diffusivity of the composite carbonates with the addition of a fixed mass fraction of graphene, and it gradually becomes larger with the increase in the addition of Al_2O_3 nanoparticles. The thermal diffusivity of the samples with the addition of multidimensional particles fluctuated less with increasing temperature, and the stability was enhanced. As shown in Fig. 8 (b), the thermal diffusivities of the composite carbonates were enhanced by 5.135%, 11.08%, 18.92%, and 22.43% at 450 °C after the addition of Al_2O_3 nanoparticles with mass fractions of 0.5%, 1%, 1.5%, and 2%, respectively, as compared to the samples with only 0.5% graphene nanosheets and as compared to the ternary carbonate-based salt, the thermal diffusivities were maximally enhanced by 54.08%. This is since two-dimensional graphene nanosheets with high specific surface area characteristics can separate zero-dimensional Al_2O_3 nanoparticles, which effectively avoids the sedimentation of Al_2O_3 nanoparticles and enhances their stability. Meanwhile, Al_2O_3 nanoparticles can be filled between the graphene nanosheets to form a more robust thermal conductivity network, which strengthens the heat transfer structure inside the composite carbonate and improves thermal diffusivity.

4. CONCLUSIONS

In this paper, based on the aqueous solvation method, composite carbonate materials with zero-dimensional alumina nanoparticles added with different mass fractions were prepared by using ternary carbonate (Li_2CO_3:Na_2CO_3:K_2CO_3 = 40%:30%:30%, wt.%) as the base salt to study the reinforcing characteristics of zero-dimensional nanoparticles on the thermo-physical properties of the carbonates, and based on which, we investigate the synergistic thermo-physical reinforcing of multi-dimensional nanoparticles The following main conclusions were obtained through experimental studies:

1) The thermal diffusivity of the nanocomposite carbonate material increases and then decreases with the increase in the mass fraction of added zero-dimensional alumina nanoparticles, and it is maximum at an added mass fraction of 1% when the thermal diffusivity enhancement is 21.92%; The thermal diffusivity of the samples with added mass fractions of 0.5% and 1.5% showed large fluctuations with temperature, and the nanoparticles were not well stabilized. A weakening of the thermal diffusivity of the nanocomposite carbonate material was observed at an added mass fraction of 2%;

2) Due to the synergistic enhancement of thermophysical properties between zero-dimensional alumina nanoparticles and two-dimensional graphene nanosheets, the maximum enhancement of thermal diffusivity of composite carbonates with the addition of 1:1 multidimensional nanoparticles in the ratio of 1:1 can reach 42.85%;

3) The addition of two-dimensional graphene nanosheets can effectively solve the problem of decreasing thermal conductivity that occurs when the concentration of zero-dimensional Al_2O_3 nanoparticles is too high. With the addition of zero-dimensional alumina nanoparticles and an additional 0.5% mass fraction of two-dimensional graphene nanosheets, the thermal diffusivity of the composite carbonate can be enhanced by a maximum of 54.08%, and the stability of the composite carbonate is improved.

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DECLARATION OF INTEREST STATEMENT

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. All authors read and approved the final manuscript.

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