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Nanoparticle-modified thermoelectric tetrahedrite as an effective nucleating agent for sodium acetate trihydrate based phase change materials

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Abstract

Sodium acetate trihydrate phase change materials (SAT-PCMs) for thermal energy storage have been studied and applied widely because of their enhanced energy efficiency and environmental friendliness, while their application on energy exchanging requires stable chemical property and great heat storage-release merit. Nanoparticles-modified tetrahedrite (CNAS-NPs) composites show high thermoelectric properties among copper sulfides, and present excellent nucleating performance in SAT-PCMs. As characterized, the NPs were uniformly distributed into the CNAS grains, and a large number of nanopores were appeared in the grain boundaries after SPS treatment. As a heterogeneous nucleation site, NPs were used to reduce the thermal conductivity, but simultaneously maintained a relatively high electrical conductivity. As a result, by adding a small quantity of SiC, a relatively high $ZT \sim 1.0$ at 723 K is obtained, which increases by $\sim 42\%$ in the pure CNAS sample. Finally, the CNAS-NPs regarded as nucleating agents obviously decrease the supercooling degree and improve the heat storage-release cycle property. These SAT-PCMs also possess high latent heat, fine chemical stability and robust nanostructures, which illustrates promising potentials in practical applications.

1. Introduction

To solve the contradiction between the increasing energy demand and the environmental pollution caused by the use of fossil fuels, seeking renewable and pollution-free energy sources has become a worldwide challenge [1, 2]. Thermoelectric (TE) materials directly enable exchange heat to electricity energy and vice versa, whose performance is defined as the figure of merit ($ZT$), $ZT = S^2\sigma T/k$, where $S$, $\sigma$, $T$ and $k$ are the Seebeck coefficient, electrical conductivity, absolute temperature, and electrical and thermal conductivity, respectively [3, 4]. To obtain a high TE performance, nano-engineering is used to reduce the lattice thermal conductivity ($k_{\text{lat}}$) while simultaneously maintains a high power factor ($S^2\sigma PF$) [5].

Introducing nanoparticles into the matrix materials seems to be an effective method to reduce thermal conductivity. The added NPs, such as SiO$_2$ [6], SiC [7, 8], MnS [9], Ag [10], BaFe$_{12}$O$_{19}$ [11] and so on, can enhance the low-middle-frequency phonon scattering. Compared with in situ nanoprecipitates [12, 13], NPs-modified composites have great advantages for decreasing thermal conductivity: the higher electrical thermal conductivity, the less influence on chemical compositions of the matrix materials, and the better chemical stability in high temperature measurement. Although they receive significant results in various TE materials, they are more or less overlooked within tetrahedrite compounds. Tetrahedrite (Cu$_{12}$Sb$_4$S$_{13}$, abbreviated as CAS) has a cost-effective merit of constitution elements, processes a complex Sb[Cu$_5$]Sb rattler structure, and consequently has received great attention in the copper sulfides [14, 15]. Compared to the TE materials of high Seebeck coefficient and zT values, such as In$_{0.2}$Ce$_{0.2}$Co$_4$Sb$_{12}$, LaTe$_{1.45}$, Zn$_{4.4}$Cd$_{0.6}$Sb$_3$, and CeFe$_{5.3}$Co$_{0.5}$Sb$_{12}$
CAS seems to be not impressive. But its low thermal conductivity, environment-friendly and cost effective nature make it attractive worldwide. Many great works reported its TE properties and obtained high ZT values through single or multiple elements doping. Sun et al [18] firstly presented a high ZT value 1.0 through introducing ZnO NPs into CAS matrix by using the method of simple mechanical alloying (MA) and spark plasma sintering (SPS), because of the strong low/mid-frequency phonon scattering from ZnO heterogeneous nucleation site. It was further discovered that adding ultralow particles of Nb2O5 displayed a state-of-the-art ZT value about 1.2 at 723 K, which was result from the largely reduced lattice thermal conductivity through the fine-grained nanostructures and grain boundary-distributed NPs in thermoelectric tetrahedrites [19]. Therefore, it has great potential to study the particle size effects on thermoelectric properties in tetrahedrites materials in details.

Simultaneously, CAS, acted as a natural mineral, seems to be a promising nucleating agent in eutectic salt-based phase change energy storage field due to its various crystal structures. By introducing NPs into the matrix, small grains and nanopores are obtained, which will provide a base for nucleation, and then reduce the undercooling. Sodium acetate trihydrate (SAT, CH3COONa · 3H2O) is a promising phase change material (PCM) and has been employed in industry for decades because of some advantages, large latent heat of fusion ~260 J g−1, low phase change temperature ~331 K, no corrosive and cost-effective elements [20–22]. So far, SAT has widely applied to solar heat storage, waste heat recovery, energy conservation in buildings and other industrial processes [23]. However, large supercooling degree as a shortcoming in SAT-based PCMs has been limiting practical applications on an industrial scale [24]. Supercooling is an intrinsic property for eutectic salt PCMs. There are rarely reliable solutions to mitigate supercooling in literatures. Many great studies have been attempting to improve the performance of SAT PCMs [25]. They focused on techniques aimed to improve the thermal conductivity, approaches limited or prevented supercooling, and solutions weaken the thermal degradation of melting salt hydrates [26]. Adding nucleating agent is an effective strategy using homogenous or heterogenous seeds. The suspended particles (especially NPs) or colloids aid in the crystal nuclei formation by reducing the critical nucleation work of crystal, and then improve the nucleation rate. This method has attracted in a great number of publications, such as introducing AlN NPs [27], α-Al2O3 NPs [28], ZrO2 nanofillers [29], chitin nanowhisker [30], nano copper [31] and nano silver [32] into SAT subtract, which presented promising results in reducing supercooling as their optimal concentration ranges. In most cases, the supercooling degree can be reduced by a few Kelvin through isomorphic nucleators. This does not always hold true, but there is an opinion that small supercooling would be always achieved through decreasing mismatch of crystal lattice. Hence, it is important to seek or prepare appropriate nucleators that effectively suppress supercooling in SAT PCMs with large latent heat storage.

Herein, a series of NPs modified Cu11.5Ni0.5Sb4S12.7 composites (CNAS-NPs) were synthesized by the method of MA + SPS. These NPs were homogeneously distributed into the CNAS grains, which had a small impact on the electrical performance. A large number of nanopores were appeared in the grain boundaries after SPS treatment, which resulted in the largely reduced thermal conductivity. As a result, a relatively high ZT value about 1.0 is obtained at 723 K for the SiC-modified tetrahedrite. Furthermore, the CNAS-NPs powder displays the fine nucleating performance in improving the supercooling degree and melting-freezing cycling property for the SAT-based PCMs. It is found that the supercooling value decreases to ~5 K as the CNAS-NPs nucleators are added.

### 2. Experimental design

#### 2.1. Chemicals and samples preparation

All chemicals were purchased from Aladdin Industrial Corporation (China), including Sb shots, powders of Cu, Ni, S, and NPs of B4C, SnO2, Diamond, SiC. These materials were weighed in the composition ratio of Cu11.5Ni0.5Sb4S12.7−0.1 vol% NPs in an Ar-filled glove box, then loaded into a stainless steel container with balls, which was filled with Ar-H2 gases. The mixture was subjected to mechanical alloying at 450 rpm for 10 hours in a planetary ball mill (QM–3SP2). The produced powders were subsequently put into a graphite die, then sintered into bulks by spark plasma sintering (SPS, SCM–1000-1) under 50 MPa axial pressure, which was at 723 K in vacuum. The bulks were cooled down to RT at the rate of 15 K min−1. Thus, a series of Cu11.5Ni0.5Sb4S12.7−δNPs (NPs = B4C, SnO2, Diamond and SiC, abbreviated as CNAS-NPs) disks with highly relative densities (above 95%) were obtained. Furthermore, the CNAS-NPs bulks were milled to powders under Ar-H2 atmosphere, in order to further carry out the heat storage-and-release process as nucleating agents in sodium acetate trihydrate (SAT) phase change material.
2.2. Structural characterization
The phase structures of synthesized CNAS-NPs samples were characterized by x-ray diffraction (XRD, D8 equipped with Ge (111) monochromator) with Cu Kα radiation. The elemental distribution of bulks was obtained by electronic probe microscopic analysis (EPMA, JXA-8230) with wavelength-dispersive spectrometer. The morphologies were observed by field emission scanning electron microscopy (FESEM, JSM-7001) and the crystal structures were conducted by transmission electron microscopy (TEM, 2100) operated at 200 kV.

2.3. Thermoelectric transport property
After cut and polished of the bar- and disk-shaped samples, the thermoelectric properties were investigated. The Seebeck coefficient/electrical resistance measuring device (ZEM-3) was used to get the electrical resistivity (ρ) and Seebeck coefficient (S) of bar-shaped bulks simultaneously at 303 K–723 K. The laser flash instrument (LFA457) was utilized to measure the thermal diffusivity (D). The thermal conductivity (κtot) was calculated by the equation of κ = DρCp, where ρ represents the density measured by the Archimedes method and Cp stands for the specific heat capacity respectively.

2.4. Heat storage-and-release property
The phase change materials (PCMs) with different mass fractions of SAT (99%), sodium carboxymethylcellulose (CMC, 1000–1400 MPa s), disodium hydrogen phosphate dodecahydrate (DSP, 99%), graphite (200 meshes) and CNAS-NPs were prepared using a low-speed ball milling process. The mass ratio of SAT: CMC: DSP: G: CNAS-NPs is 98: 2: 1: 10:1, and a series of PCMs abbreviated PCMs-NPs (NP = B4C, SnO2, Diamond and SiC). Their heat storage/release properties were studied in a heat storage setup, which was reported in [22, 33] in detail. Simply, the PCMs-NPs samples were sealed in the test tubes, placed in the water bath as temperatures increased from indoor temperature (∼300 K) to 336 K, then naturally cooled down. The DAQ970A (Keysight Technologies) was used for real-time data acquisition at entire testing processes.

3. Results and discussion
3.1. Thermoelectric properties of CNAS-NPs compounds
Figure 1 shows the thermoelectric properties of CNAS-NPs (NP = B4C, SnO2, Diamond and SiC) composites synthesized by a MA-SPS method. Compared to the Na0.95Pb20SbTe22, which has a zT = 0.36 W m⁻¹ K⁻¹, the zT values for all CNAS-NPs composites are increased from 5.92 to 4.15 × 10⁻⁵ m³⁻¹ at 573 K as added B4C. The reduced σ values largely derive from the electrical insulation NPs and the strong electrons scattering of NPs, as compared with previous reports [34]. The Seebeck coefficient (S) increases linearly against temperature, which exhibits typical degenerate conductor behaviors. As indicated, large particle size of B4C (discussed in detail later) yields larger S than that of other NPs and pure-phased samples at entire temperatures, suggesting the decreased carrier concentration and additional scattering effects. The maximal S value is 178 μV K⁻¹ for CNAS-B4C at 723 K. Compared with NPs-free sample, as shown in figure S1, adding small particle size of SiC (CNAS-SiC) possesses almost the same power factor (PF). The maximal PF value is 12.6 μW cm⁻¹ K⁻² at 723 K. Other NPs modified CNAS samples show the small PF due to the large reduced electrical conductivities.

The measured Cp indicates that the κtot is quite low (less than 1.0 W m⁻¹ K⁻¹) for all CNAS-NPs composites at the temperature range from 303 K to 723 K compared to porous Si, which is 7.6 W m⁻¹ K⁻¹ [35]. It is the phonon scattering from the special microstructures that reduce the thermal conductivity. The Wiedemann–Franz law [36] is used to remove the electric contribution from the κtot values, by which lattice thermal conductivity (κlat) values are gained. The Lorenz number (in figure S2) can be firstly obtained via the assumption of transport, which is dominated by acoustic scattering and single parabolic band. Based on the Debye–Callaway’s equation [37], nanostructures (nanopores, nanoparticles and nanopinances) phonon-scattering with a relaxation time rely on the frequency as τ ∼ ω⁰ + ω⁻¹ + ω⁻³ target the low-middle frequencyphonons. Indeed, at high temperatures (above 523 K, in figure 1(c)), the nanostructure-effect seems more sensitive to phonons contribution, which leads to a large reduction of κlat from B4C to SiC. The lowest κlat value is 0.36 W m⁻¹ K⁻¹ for CNAS-SiC bulks at the temperature of 673 K.

Combined with the PF and largely reduced κlat, a high-performance thermoelectric property is obtained for CNAS-NPs composites at a temperature range from 303 to 723 K, as shown in figure 1(d). The highest ZT value of 0.98 is achieved at 723 K for CNAS-SiC sample, which is increased by ~40% for NPs-free sample.
3.2. Structure characteristics of CNAS-NPs compounds

The enhanced thermoelectric performance is derived from the special microstructures for CNAS-NPs composites. Therefore, the merits contained micromorphology, crystal structures and elemental distributions are measured and discussed in detail. In the study, these introduced nanoparticles have wide size ranges from 400 nm \(B_4C\), figure 2(a)) to 40 nm \(SiC\), figure 2(d)), and present prefect crystallinities. Adding NPs in tetrahedrites reveals no significant differences among XRD patterns (figure 3(a)), indicating no other than the Cu\(_{12}\)Sb\(_4\)S\(_{13}\) matrix with \(I\bar{4}3\)\(_m\) symmetry. No peaks related to NPs are detected because of the low content and good distribution. Furthermore, as shown in figure 3(b), SiC modified CNAS sample exhibits a single-phase structure from refinement XRD result.

Figure 4(a) shows the micromorphology of CNAS-SiC sample on fractured surface. Small grain size with \(\sim 1.0\) \(\mu\)m is easily observed, and nanopores of \(\sim 100\) nm are also induced by introducing nano-SiC particles. The Cu, Sb, S, Ni and Si NPs are uniformly distributed into the CNAS substrate, as displayed in figures 4(b)–(f). In order to further confirm the morphologies and distributions of SiC NPs in the bulks, TEM measurement was carried out for CNAS-SiC sample, and the results were presented in figure 5. At first sight, small grain sizes of ca. 1 \(\mu\)m and nanopores are obtained as introducing SiC NPs in the Cu\(_{12}\)Sb\(_4\)S\(_{13}\) matrix, which is well consistent with SEM result in figure 4(a). The SiC NPs (white solid rectangles, figure 5(a)) are observed surrounding the CNAS grains, and the number of nanopores (white dash cycles) noticed in the triple junctions, which is caused by the different thermal diffusivity between the NPs and the matrix during SPS treatment. Interestingly, it is found that the decompositions and grain growth of SiC are not happened after SPS treatment. Figure 5(b) exhibits the details of the interfaces originating from figure 5(a), which shows a clear grain boundary between the CNAS and the SiC (marked with white dash line). Fine high-resolution TEM (HRTEM) images of free of defects in the (1\(\bar{1}\)0) and (200) planes for CNAS are kept along the [001] direction, as shown in figure 5(b) and its inset EDS figure. However, the SiC NPs present a very dark image, and unable to further study its crystal structures because of the excellent mechanical property during the Ar ions etching. As concluded from XRD, SEM, EPMA and TEM, the fine nano-structures could improve the electrical performance for CNAS-SiC composite, which is well agree with the results in figure 1(a).
3.3. Heat storage-release behavior of PCMs-NPs composites
Sodium acetate trihydrate with relatively low phase change temperature of \( T_m \sim 331 \text{ K} \) exhibits excellent latent heat capacity, and attracts wide attention in phase change heat storage field. Correspondingly, the melting-solidification mechanism can be expressed \([38]\):

\[
\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O} \rightleftharpoons \text{CH}_3\text{COONa} \cdot n\text{H}_2\text{O} + (3 - n) \text{H}_2\text{O}
\]  

(1)

When temperature above \( T_m \), SAT presents phase change as absorbed much heat, and lose 3-\( n \) molar of water molecules (formula (1)). As SAT cooled under \( T_m \), the absorbed heat will be released, and then the phase separation is happened at room temperature (RT) because of the different density between \( \text{CH}_3\text{COONa} \cdot n\text{H}_2\text{O} \) crystals and liquid water. During nucleation, the chemical potential of water increases. As the water molecules rearrange themselves into the surroundings of the solid hydrate, the chemical potential of water in crystalline state, \( \mu_c \), is higher than that in the supercooled melt state, \( \mu_m \). The difference of the chemical potential

\[ \Delta \mu = \mu_c - \mu_m \]
may be considered a rough measure of the barrier to nucleation [39]. Finally, the supercooling degree \( \Delta T_s \), sometimes as large as \( \sim 30 \) K, is generated.

Phase separation and supercooling obviously suppress the latent heat release for pure SAT [40, 41]. As shown in figure 6, however, adding CNAS-NPs nucleating agents effectively reduced \( \Delta T_s \). As listed in table 1, the \( \Delta T_s \) values dropped to about 5 K (except for PCMs-SnO\(_2\) sample). Figure S3 present the microstructures of PCMs-NPs after 100 heat storage-release cycles, showing uniform crystals and large number of pores, which indicates the fine mechanical performance for PCMs-NPs composite. The curves of \( \Delta T_s \) dependent on melting-freezing cycles are displayed in figure S4, and the corresponding thermal transport parameters are listed in table S1. The \( \Delta T_s \) values of PCMs-NPs sample are less than 6 K even 100 cycles, and possesses the high thermal
properties. The PCMs-NPs have great merits of high latent heat, small supercooling degree and uniform microstructures, thereby, showing an excellent potential in practical application.

4. Conclusions

In this study, nanoparticle modified tetrahedrites were synthesized by the method of MA + SPS. Nanoparticles were homogeneously distributed into the CNAS matrix, and a great number of nanopores could be observed along the grain boundaries. Introducing NPs largely reduces the thermal conductivity of CNAS-NPs composites because of the strong low-middle frequency phonon-scattering, meanwhile maintaining a relatively high electrical conductivity. The lowest lattice thermal conductivity of ∼0.36 W m⁻¹ K⁻¹ is obtained for CNAS-SiC sample. Finally, a relatively high ZT value about 1.0 is achieved at the temperature of 723 K, which increases by ∼42% for the pure-phased sample. Furthermore, CNAS-NPs as an excellent nucleating agent can effectively suppress the phase separation and decrease the supercooling in sodium acetate trihydrate-based PCMs. A small ΔTₛ value, ∼6 K, is obtained even after 100 heat storage-release cycles. Additionally, the PCMs-NPs composites present robust crystals under measurements, which indicate a fine mechanical property. The PCMs-NPs materials with the great merits of high latent heat, small supercooling degree and excellent chemical stability display huge potentials in phase change heat storage applications.

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Competing interests

The authors declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.
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