

PREPARATION OF CARBON NANOTUBE-BASED FLEXIBLE PHASE CHANGE FILMS AND THEIR APPLICATION IN THERMOPHOTOVOLTAIC CONVERSION

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Abstract: *With the growing demand for renewable energy, the application of thermophotovoltaic conversion materials in energy storage and conversion has attracted significant attention. To enhance thermophotovoltaic conversion efficiency and promote the development of energy storage technologies, this study introduces modified carbon nanotubes (CNTs) to improve the thermal conductivity of flexible phase change films made from polylactic acid (PLA) and polyethylene glycol (PEG), and investigates their shape stability, phase change properties, thermal conductivity, and thermophotovoltaic conversion performance. The results show that the CNT-modified films exhibit excellent shape stability and flexibility, maintaining their shape above the melting temperature of PEG and recovering to their original form upon cooling. As the CNT content increases, the thermal conductivity of the films significantly improves, with the 7% CNT composite film showing a 158% increase in thermal conductivity, although the latent heat value decreases slightly. The thermophotovoltaic conversion efficiency increases with the CNT content, rising from 79.3% (1% CNTs) to 91.8% (7% CNTs), while the phase change time is reduced. The introduction of CNTs optimizes the heat transfer process, significantly enhancing the thermophotovoltaic conversion performance of the films. This study provides a promising solution for the development of solar thermal storage materials.*

Keywords: *Flexible phase change films; Thermophotovoltaic conversion; Phase change; Thermal energy storage.*

1. Introduction

The utilization of solar energy is of increasing interest with the heightened concern for the development of low carbon industries, population growth and fuel depletion[1, 2]. Among the various forms of solar energy applications, solar thermal conversion, i.e., the collection of solar radiation and its direct conversion into thermal energy, is considered one of the most effective and reliable methods for harnessing the abundant solar energy[3-5]. The superior storage capacity and near-constant operating temperature of latent heat storage in phase change materials make PCMs a promising material for solar

thermal storage[6, 7]. However, PCMs also suffer from disadvantages such as poor thermal conductivity, liquid leakage, shape instability, and poor mechanical properties during practical use[8, 9].

To improve the thermal conductivity of PCMs, Wu et al. [10, 11] enhanced the thermal conductivity of paraffin by adding graphene nanosheets (GNPs) and carbon nanotubes (CNTs) into the PCM. To address issues such as liquid leakage in PCMs, researchers have achieved leak-proof and improved flexibility by microencapsulating the phase change material or encapsulating it within the 3D porous structure of high melting point polymers [12-14]. Microencapsulated PCMs have overcome the limitations of traditional single-phase phase change materials, but their low thermal conductivity significantly limits their applications[15]. To further enhance the thermal conductivity of microcapsules, high thermal conductivity nanoparticles or surface modification techniques can be used to improve their heat transfer properties. Wu et al. [16] used melamine-formaldehyde resin as the wall material and incorporated high thermal conductivity materials such as graphite, graphene, and expanded graphite into paraffin. They prepared modified phase change microcapsules via an in-situ polymerization method, which significantly improved the thermal conductivity of the material. Polymers represented by polyethylene, polyurethane and thermoplastic elastomers are widely used to prepare shaped flexible phase change materials, which are composite phase change materials with stable phase change temperature and good flexibility and can be made into different shapes according to the demand[17-21]. However, polymers are difficult to degrade and easily cause damage to the environment after disposal, which does not meet the future development trend of greening. Biodegradable materials are gradually used in daily life with their safe and non-toxic, biodegradable properties, but their applications in industrial fields are still in the development stage. Poly(lactic acid) (PLA) is a biodegradable polyester with good processability and environmental friendliness [22], and it has been compounded with phase change materials by researchers. Lu et al. [23] prepared the first definitive phase change material by melt blending method using PLA as the support substrate and HDPE as the phase change workpiece. However, PLA is more ductile compared to other polymeric support materials, and polyethylene glycol (PEG) is often used as a plasticizer to improve the ductility of PLA[24, 25]. Meanwhile, polyethylene glycol has become a common functional filler for flexible phase change materials as an organic phase change material with the advantages of high latent heat and environmental economy[26-28].

In the field of photothermal conversion of phase change materials, in addition to high phase change enthalpy, they should also have high light absorption, and carbon-based materials with full-spectrum absorption properties and good stability are more widely used[29-31]. The synthesis of flexible shaped phase change materials with excellent thermal and photothermal conversion properties is one of the hot issues in current research by giving the phase change materials light absorption and photothermal conversion capabilities, improving the thermal performance of the shaped phase change materials, and reducing the interfacial thermal resistance between the carrier and the phase change core material. In this paper, carbon nanotubes are modified with silane coupling agent and introduced into PEG/PLA flexible phase change films, and tributyl citrate is added to enhance the flexibility of the films, which gives the flexible phase change films photothermal conversion properties and further enhances their thermal conductivity, and their thermal properties, thermal conductivity and photothermal conversion properties are investigated.

2. Experimental

2.1. Materials

PEG(phase change temperature range 51-54°C,2000), purchased from Shanghai Maclean Company; PLA, purchased from Nature Works; Methylene chloride as solvent, purchased from Tianjin Fuyu Fine Chemical Company; Silane coupling agent as modifier, purchased from Jinan Feilong Chemical Company, a bridging molecule that bonds to inorganic surfaces via silanol groups while connecting to polymers through organic functional groups; Carbon nanotubes, purchased from Shanghai Maclean Company; Tributyl citrate as plasticizer, purchased from Shanghai Maclean Company McLean Company. Tributyl citrate was selected as a flexibilizer, whose flexible aliphatic long chains can intercalate between PLA molecular chains. By reducing intermolecular forces (van der Waals interactions) and increasing free volume, it significantly enhances the material's flexibility.

2.2. Preparation of modified carbon nanotubes

First, 0.5g CNT was placed in the mixed acid of 60 ml concentrated sulfuric acid and concentrated nitric acid (3:1). After stirring evenly with a stirring rod, it was shaken in a 50°C ultrasonic instrument for 10h and diluted with a lot of deionized water. Then use an aperture 0.22 μm membrane filter for filtering and washing to neutral, then put in a vacuum drying oven drying the product to get product b. The product b was dissolved in 200 mL anhydrous ethanol for ultrasonic dispersion for 30 min, then 10 mL silane coupling agent was added into the carbon nanotubes solution, and then centrifuged in a water bath for 4000 r/min for 10 min. The upper liquid was removed to obtain black precipitate, and the final product was obtained after drying for 12h.

2.3. Preparation of flexible phase change films of carbon nanotubes

First, PLA granules were dried in a blast drying oven at 70°C for 12h and PEG at room temperature for 12h to remove moisture. Then, 5g PLA and 9g PEG were weighed proportionally, respectively, and the modified CNTs were weighed at the proportions of 1%, 3%, 5% and 7%. The corresponding mass was shown in Tab. 1. After that, CNTs were poured into 50 ml DCM solution, covered with plastic wrap to prevent solvent volatilization, placed in an ultrasonic shaker for 30 min, and dispersed evenly. After that, PLA and PEG were added in turn, weighed and then placed on a magnetic agitator for 2h at room temperature, and 2 ml TBC was added as a plasticizer during the stirring process. After the stirring, the casting liquid was spread on the glass petri dish by the casting method, placed at room temperature for 12 h, and then dried in a drying oven for 6 h, so that DCM was fully volatilized, and CNT flexible phase change film was prepared.

Tab.1 Ratio of CNT flexible phase change films

Samples	m _{PEG} (g)	m _{PLA} (g)	m _{CNTs} (g)	CNTs content
CPCF-1	9	5	0.14	1%
CPCF-2	9	5	0.43	3%
CPCF-3	9	5	0.74	5%
CPCF-4	9	5	1.05	7%

3. Results and discussion

3.1. Chemical structure characterization of carbon nanotubes before and after modification

To study the changes of functional groups before and after the modification of carbon nanotubes, their FT-IR infrared spectrograms (as in Fig.1) were investigated. After modification with silane coupling agent, a peak was generated at 1063.38 cm^{-1} , which proves that carboxyl groups were formed on the acidified CNTs, which reacted with KH-550 to form C-O-Si bonds, the peak near 2981 cm^{-1} is due to the appearance of a new methylene ($-\text{CH}_2-$) stretching absorption vibration peak, which is present on KH-550, and the peaks near 1244 cm^{-1} and the peaks near 1397 cm^{-1} are C=O bond characteristic absorption peaks, which indicate that the carboxyl group has been successfully grafted onto the carbon nanotubes, and the carboxylated carbon nanotubes contain carboxyl groups compared to the unmodified carbon nanotubes, through the modification treatment, the surface layer of the carbon nanotubes gets hydrophilic groups, which facilitate their better dispersion within the solvent. Fig.2 shows the XRD spectra of carbon nanotubes before and after modification, from which it can be seen that the CNTs have the same diffraction peaks before and after modification, indicating that the crystallinity of the CNTs is not affected before and after modification, and the phase composition of the CNTs themselves is not changed. The increased intensity of the diffraction peaks of the modified CNTs was mainly due to the successful grafting of Si and O elements on the silane coupling agent KH-550 to the CNTs, and this conclusion was consistent with the results of FT-IR spectroscopy.

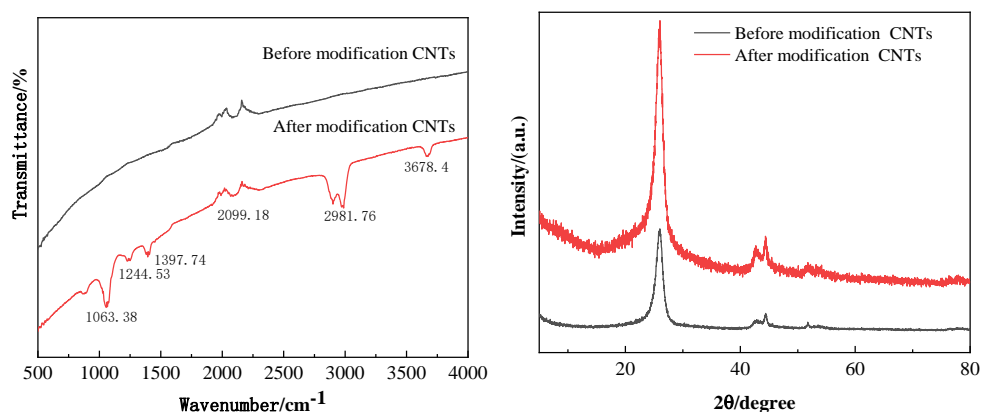


Fig.1 FT-IR spectra of CNTs before and after modification Fig.2 XRD patterns of CNTs before and after modification

3.2. Carbon nanotube flexible phase change film flexibility and its deformation phase change properties

The film was bent, folded and stretched, and the results are shown in Fig.3a. It can be observed that the film still has good flexibility after the addition of carbon nanotubes and can be folded at will without fracture. Due to the addition of TBC to plasticize it, the film does not break after stretching, which proves that the carbon nanotube flexible phase change film has good flexibility. Meanwhile, the acidification treatment opens the C-C bond in CNTs and generates carboxyl groups, which helps to improve the dispersion of CNTs, thus effectively enhancing the mechanical properties of the material. The film was cut into $3\times 3\text{ cm}^2$ pieces, and a certain mass of PEG2000 was put into a constant temperature oven at 80°C at the same time, and removed after five minutes to observe the leakage, and the results

are shown in Fig.3b. PEG melted into liquid state after heating for 5 min, but the film remained intact and did not leak. This was due to the good compatibility between PEG and PLA, the compatibility between PLA and PEG primarily stems from intermolecular hydrogen bonding formed between the carbonyl oxygen atoms (C=O) of PLA and the terminal hydroxyl groups (-OH) of PEG. The PEG was confined in the mesh PLA structure, which limited its liquid flow, and the addition of alumina did not affect its shaping function, and the prepared carbon nanotube flexible phase change film still had good shape stability. As can be seen from Fig.3c, the shape of the film can still be maintained above the melting temperature of PEG, and when the room temperature is restored, the film regains its original shape, proving that the phase change film still has good flexibility over a wide range of temperatures.

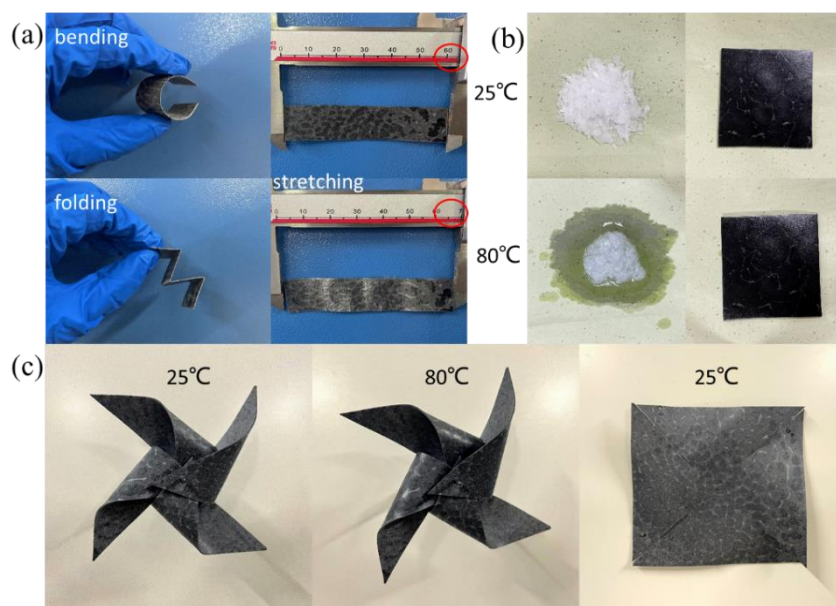


Fig. 3 Test of the flexibility and formable phase change performance of CNT flexible phase change thin films

3.3. Morphological characteristics analysis

SEM images can be used to evaluate the microscopic morphology of composite phase change materials, and the surface morphology as well as the internal structure of the material can be analyzed by observing the microscopic morphology of its surface and cross-section. Fig.4a show the SEM images of the surface of the flexible phase change films of carbon nanotubes, and it is observed that the surface is relatively smooth, which proves that the films prepared by the solution blending method have a good flatness. Meanwhile, the addition of CNT will realign the mobility of the PLA chain and make the PLA chain more regular. Fig.4b show the cross-sectional images of the CNT flexible phase change films, which can be seen to have a similar structure to that of the alumina-added films, with the formation of a pore-like structure, which is also caused by the formation of channels within the films due to solvent volatilization. It can be seen from the figure that the phase change film prepared by solution blending method has a good deformation effect. The brighter three-dimensional mesh structure and the gray area in the figure correspond to the PLA mesh structure substrate and PEG phase change material, respectively, and PEG is wrapped in the mesh structure of PLA, and the fusion of the two has no obvious demarcation, which proves that PLA and PEG have good compatibility. The addition of carbon nanotubes does not change the internal structure of the film. The tubular 3D structure of carbon

nanotubes can be seen in Fig.4c, which has a higher specific surface area relative to alumina, thus making it easier to form a thermally conductive network. After local magnification of the film section by a factor of 10,000 (as shown in Fig.4d), a tubular CNTs structure appears, demonstrating that CNTs are successfully encapsulated in the PEG/PLA composite phase change material.

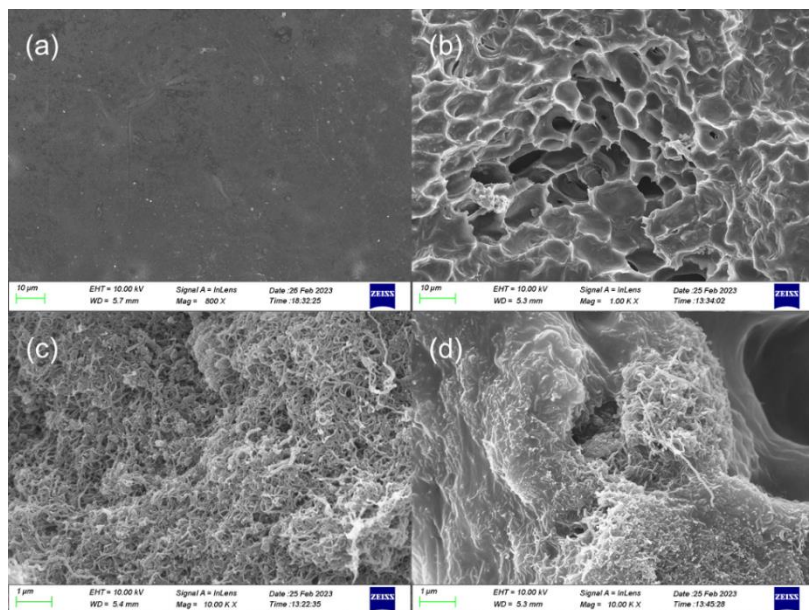


Fig.4 SEM images of (a) Surface of CNTs flexible phase change film (b) cross-section of CNTs flexible phase change film (c) Local amplification of CNTs(d)Internal film amplification

3.4. Thermal properties

Differential scanning calorimetry (DSC) is used to study the thermal energy storage properties of composite phase change materials. Fig.5(a~b) shows the heat absorption and exothermic curves of the flexible phase change film, and the phase change enthalpies in the melting (upward curve) and solidification processes (downward curve) of the film are obtained from the area between the exothermic curves, and the relevant parameters are listed with Tab. 2. The DSC test results show that the melting temperature of PEG/PLA is around 54°C, which is consistent with the melting point of PEG of 51~54°C, indicating that the composite of PEG and PLA does not change the phase change temperature of the phase change material, and with the addition of CNT, the composite phase change material maintains a melting temperature comparable to pure PEG while exhibiting reduced phase change latent heat. Heat in the polymer matrix propagates primarily through phonons with varying vibrational frequencies, and the higher the content of CNTs the lower its latent heat value. This is because the proportion of effective phase change components is reduced. At the same time, the addition of CNTs affected the crystallization of PEG, which led to a decrease in the enthalpy value.

To demonstrate the thermal stability of CNTs flexible phase change films, the CPCF-3 flexible phase change film samples were tested by DSC in cold and hot cycles. The 500 thermal cycles test is designed to simulate the long-term stability of phase change films in practical applications, evaluating their performance degradation and structural reliability after repeated phase transitions. Fig.5c shows the DSC curves of the samples before and after 500 cycles of cold and heat between 20°C and 80°C. The phase transition behavior was recorded every 100 cycles, and it was found that the phase transition temperature of the samples did not change significantly after 500 cycles of cold and heat, and the latent

heat of fusion of the samples changed from the original 104.6 J/g to 101.7 J/g, which was only about 2.8% lower than the original latent heat, proving that the flexible phase transition of carbon nanotubes. This proved that the carbon nanotube flexible phase change film has good thermal stability and reusability.

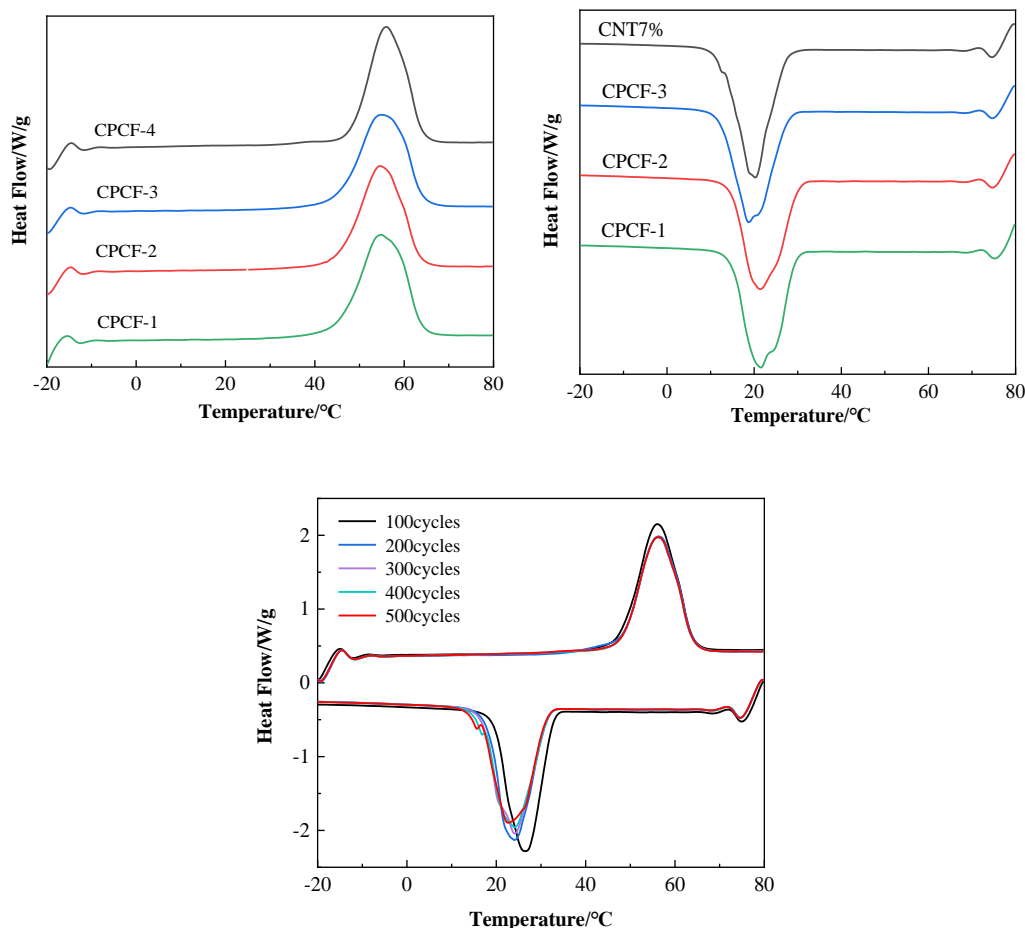


Fig. 5 (a) Endothermic DSC curve of CPCF-X thin film (b) exothermic DSC curve of CPCF-X thin film (c) Cycles of DSC curves of CPCF-X thin film

Tab. 2 DSC data of flexible phase change films

Samples	ΔH (J/g)		T_{peak} (°C)		T_{on} (°C)		T_{end} (°C)	
	M	S	M	S	M	S	M	S
PEG	193.3	190.1	54.15	24.26	49.04	16.67	60.85	28.82
CPCF-1	111.3	113.1	53.99	22.63	45.38	14.9	63.73	29.35
CPCF-2	107.9	109.5	53.77	22.36	45.45	14.81	63.67	29.99
CPCF-3	106.9	106.9	55.63	19.78	45.39	11.85	63.8	27.6
CPCF-4	104.6	106.3	54.51	17.89	45.89	10.09	64.29	26.68

T_{peak} : peak temperature ; T_{on} : onset temperature ; T_{end} : end temperature. M: melt process S: solidification process.

3.5. Thermal conductivity analysis

Thermal conductivity plays an important role in the conversion of light energy into heat energy and its timely storage by composite phase change materials. The magnitude of thermal conductivity is determined by the phonon propagation rate[32]. The thermal conductivity of the samples was tested by the hot-wire method. Firstly, the specific heat of the samples was tested by differential scanning calorimetry at a temperature range of 25-30°C. Secondly, the thermal diffusion coefficient of the samples was determined by thermal conductivity calorimetry with a set temperature range of 25-30°C. The thermal conductivity of the film is determined by Eq. (1) and the relevant parameters are shown in Tab. 3.

$$\lambda = \rho C_p a \quad (1)$$

Tab. 3 Measurement parameters of thermal conductivity

Samples	C_p (J/kg·°C)	P (g/cm ³)	a (m ² ·s)	λ (W/(m·K))
CPCF-1	1.184	0.259	1.7209	0.5277
CFCF-2	1.188	0.266	1.7459	0.5517
CPCF-3	1.19	0.271	1.7518	0.5649
CPCF-4	1.193	0.276	1.8877	0.6216

Carbon nanotubes significantly enhance the thermal conductivity of composite materials by forming a three-dimensional thermal network, thereby accelerating the phase transition process. Fig.6 shows the histogram of the thermal conductivity of the film at 25°C for the CNTs ratio. The thermal conductivity of 1%, 3%, 5% and 7% CNTs added at 25°C were 0.52 W/(m·K), 0.55 W/(m·K), 0.56 W/(m·K) and 0.62 W/(m·K), respectively, which improved the thermal conductivity by 116%, 129%, 133% and 158%, respectively, compared with the films without CNTs added. It can be seen that the thermal conductivity of the film gradually increases with the increase of CNTs content, which is due to the fact that in the polymer matrix, heat propagates mainly by phonons of different vibrational frequencies, and the inherent high thermal conductivity of CNTs and their nano-network channels can build longer heat transfer paths in the phase change materials, increasing the free range of phonon transfer and reducing the interfacial thermal resistance, which, together with their higher specific surface area, results in a faster heat transfer rate[33].

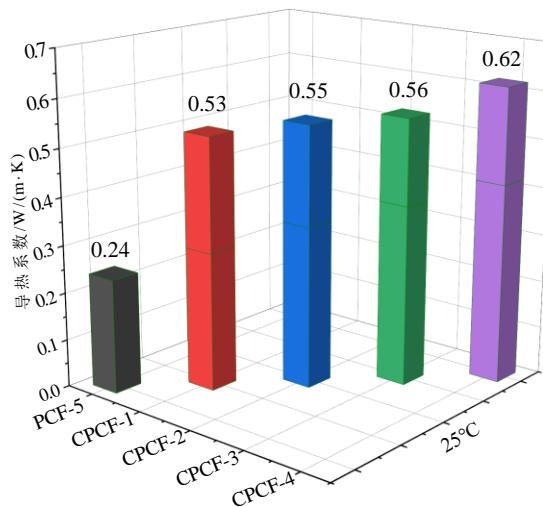


Fig. 6 Thermal conductivity of flexible thin film

3.6. Solar thermal conversion properties

To test the photothermal conversion performance of thin films, the photothermal conversion behavior and temperature change of CNTs flexible phase change films under simulated light intensity of 150 mW/cm^2 were investigated. As CNTs are extremely light-absorbing, the sunlight is instantaneously absorbed under the irradiation of simulated light source, while the photothermal conversion occurs inside the film and the thermal energy is stored in the PCM system. The photothermal conversion curves are shown in Fig.7, and the surface temperature of the composite phase change films all show an increasing trend with the increase of light exposure time. The CNTs flexible phase change film can be warmed up to 63.8°C . When the temperature reaches near the phase change temperature, a thermal storage phase change platform appears, at which time, the solid-liquid phase change occurs in the PEG inside the film and absorbs heat with almost constant temperature, and after the phase change ends, the film temperature continues to rise until the CNTs absorb light and saturate. Similarly, an exothermic phase transition plateau appeared during the cooling process, and the heating and cooling rates gradually increased with the increase of CNTs. In contrast, the PEG/PLA films only warmed up to 43.2°C . The PEG/PLA film's minimal temperature rise originates from its extremely low light absorption rate, forming a stark contrast with the active photothermal conversion of CNT composite films, thereby confirming its lack of photothermal capability. Since the PEG phase transition temperature was not reached, no phase transition plateau appeared, which proved that the PEG/PLA films did not have photothermal conversion properties and the temperature rise was caused by the thermal effect of the simulated light source. The photothermal conversion properties of thin films are derived from CNTs, which contain a large number of conjugated π -bonds, and light can be excited from π -orbital to π -orbital by a lower input energy. Therefore, a lower energy can complete the jump between π orbitals and at the same time redshift the absorption spectrum, so the conjugated π bonds in CNTs can cause the excitation of electrons at almost all wavelengths of solar visible light, and the electrons are thermally relaxed through the electron-phonon coupling mechanism[34]. The released heat energy is absorbed and stored through the PEG solid-liquid phase change, and the stored heat energy is released spontaneously after the light ends, thus completing the conversion of light energy to heat energy.

Fig.8 showed the trend of photothermal conversion efficiency and phase transition time for different CNT additions. The photothermal conversion efficiencies of the films were 79.3%, 84.9%, 88.6%, and 91.8% when the contents of CNTs were 1%, 3%, 5%, and 7%, respectively, and it can be seen that the photothermal conversion efficiency of the films gradually increased with the increase of CNTs content, while the phase change time of the films gradually decreased, which was due to the fact that the more CNTs content, the faster its heat conduction rate, which led to the intra-film PEG phase transition rate is accelerated and the phase transition time is shortened.

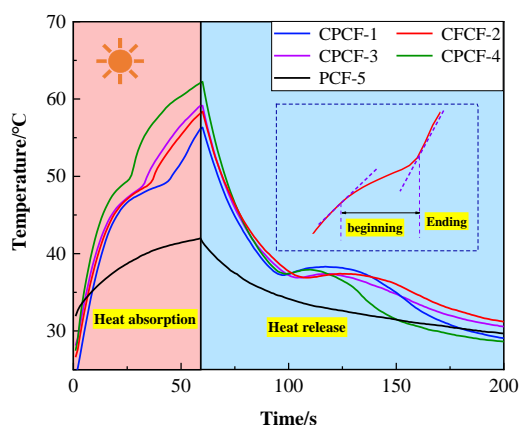


Fig.7 Curve of photothermal conversion and thermal energy storage of carbon nanotubes flexible phase change thin films

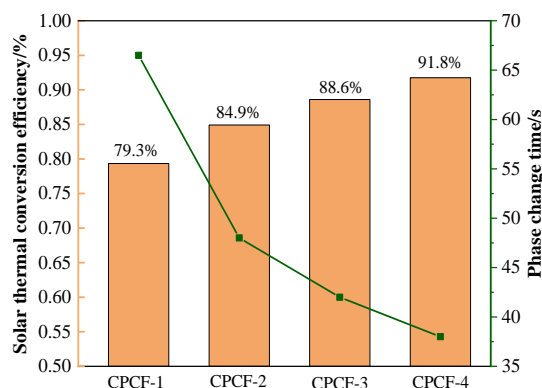


Fig.8 Variation trend of photothermal conversion efficiency and phase transition time under different CNT dosage

4. Conclusions

In this work, we introduced modified CNTs into flexible phase change films to enhance their thermal conductivity while imparting photothermal conversion properties to the films. The shape stability, phase change properties, thermal conductivity and photothermal conversion ability were investigated. By modifying CNTs with silane coupling agent, the surface layer of carbon nanotubes got hydrophilic groups, which facilitated their better dispersion in the solvent, and the physical properties of CNTs did not change before and after modification. The shape of the film remained unchanged above the melting temperature of PEG, and when the room temperature was restored, the film regained its original shape, which proved that the phase change film still had good flexibility in a wide temperature range. the DSC test results showed that the latent heat retention rate of PEG/PLA film was 62.2% and 67.6%, respectively. With the increase of CNTs content, the latent heat value of the phase change films decreases, but the thermal conductivity is effectively improved, and the addition of 7% CNTs can increase the thermal conductivity of the films by 158%. And the PEG/PLA/CNTs composite film has good reusability and thermal stability The introduction of CNTs endows the flexible phase change film with efficient photothermal conversion capability. When the temperature reached near the phase transition temperature, the thermal storage phase transition plateau appeared in the photothermal conversion process of the flexible phase change films of carbon nanotubes, and the PEG/PLA films only warmed up to 43.2°C without the phase transition plateau. the photothermal conversion efficiencies of the films were 79.3%, 84.9%, 88.6%, and 91.8% when the contents of CNTs were 1%, 3%, 5%, and 7%, respectively, and as the CNTs content increased, the photothermal conversion efficiency of the films gradually increased, while the phase transition time of the films gradually decreased.

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