

Investigation on crystallization of TiO₂–water nanofluids and deionized water

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ARTICLE INFO

Article history:

Received 19 October 2010

Received in revised form 5 July 2011

Accepted 11 July 2011

Available online 6 August 2011

Keywords:

Nanofluid

Crystallization temperature

Latent heat

Half crystallization time

DSC

TiO₂

ABSTRACT

Two titania–water (TiO₂–water) nanofluids were prepared by dispersing TiO₂ nanoparticles in rod shapes (rutile TiO₂) and in spherical shapes (anatase TiO₂) into deionized water. The effect of the TiO₂ nanoparticles on the crystallization behaviors of the nanofluids were investigated using differential scanning calorimetry (DSC). The weight fraction of the TiO₂ nanoparticles was 0.05%, 0.30% and 0.70%. The experimental results show that in the cooling rate range of 1.5–9.0 °C/min, the nanofluids with rutile TiO₂ nanoparticles had higher crystallization temperatures compared with deionized water and the nanofluids with anatase TiO₂ nanoparticles, and the heat of crystallization of the rutile TiO₂ nanofluid was larger than that of the anatase TiO₂ nanofluid. It is found that the rutile TiO₂ nanofluid is more suitable than the anatase TiO₂ nanofluid for ice storage system under the experimental conditions. Moreover, some unexpected data were obtained, which show that the shapes of the crystallization temperature curves of the nanofluids were like symbols of inverse “N” at 0.5–9.0 °C/min, and that the crystallization temperatures of some of the nanofluids were lower than that of the deionized water at low cooling rates. The mechanisms for the unusual phenomenon were discussed.

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1. Introduction

Latent heat thermal energy storage technique has proved to be more applicable to larger amount of energy storage per unit volume than sensible heat thermal energy storage [1]. Ice storage air-conditioning system is an important element of many industrial and commercial energy conservation programs. For example, the problem of imbalance of electrical demand in summer in many countries could be solved by running refrigerators to store nighttime power in ice during night [2]. Water is widely used as the phase change material for ice storage due to various advantages such as high latent heat, low cost and easy acquisition, no environmental pollution concern and compatibility with the material of cooling storage equipment. However, a degradation of the coefficient of performance of the system is caused by the low crystallization (freezing) temperature (large supercooling degree) of water during solidification process. The supercooling degree can be reduced greatly by adding some small particles into water [3,4]. Therefore, the aqueous nanofluid (nanoparticle suspension in water) would be a potential material to improve the performance of ice storage system.

Differential scanning calorimetry (DSC) is a professional instrument to study the crystallization behavior of materials, and it has been applied to the measurement of nanofluids in recent years. Zhou and Ni measured the specific heat of water-based alumina (Al₂O₃) nanofluids [5]. Xie et al. investigated the phase transitions of nanofluids containing Al₂O₃ or titania (TiO₂) nanoparticles with different weight fractions [6]. The latent heat storage capacity for 0.1% Al₂O₃–H₂O nanofluids and the heat of fusion of 1% anatase TiO₂–water nanofluid were measured to be 313.1 kJ/kg [7] and 284.7 kJ/kg [8], respectively. However, there are few studies focusing on the crystallization behaviors of nanofluids with nanoparticles of different shapes. In this study, the influences of TiO₂ nanoparticles in rod shapes and in spherical shapes on the crystallization behaviors of deionized water were investigated using DSC. The effects of cooling rate and weight fraction of nanoparticles were also analyzed.

2. Material and methods

2.1. Materials

Two TiO₂–water nanofluids were prepared, by dispersing TiO₂ nanoparticles in rod shapes (Hefei Jiankun Chemical Industry Co. Ltd., China) and in spherical shapes (Shanghai Huzheng nanotechnology Co. Ltd., China), respectively, into deionized water. The TiO₂–water nanofluids were prepared as follows. The PH value of the deionized water was adjusted to 8 using ammonia. The TiO₂ nanoparticles were added into the deionized water, with weight

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fraction of 0.05%, 0.30% and 0.70%. Sodium dodecyl sulfate (SDS, $C_{12}H_{25}SO_4Na$, analytical reagent, Guangdong Xilong Chemical materials Co. Ltd., China) was used as surfactant to ensure better stability and proper dispersion. The mass ratio of TiO_2 : SDS was 1:1. The TiO_2 and the surfactant were weighed by an electronic balance whose precision is ± 0.0001 g. The suspension was whisked by a magnetic stirring apparatus (Type 95-1, Shanghai Sile Appliance Co. Ltd., China) for 10 min and sonicated by an ultrasound generator (20 kHz, 100 W, Kejin Ultrasonic Equipment Factory of Panyu Guangzhou, China) for 40 min.

The TiO_2 nanofluids were investigated by a transmission electron microscope (TEM, JEM-1200EX) as shown in Fig. 1. The average size of the rutile TiO_2 and anatase TiO_2 nanoparticles is $20\text{ nm} \times 50\text{ nm}$ and 15 nm , respectively.

The stabilities of the TiO_2 -water nanofluids can be estimated by the changes in the weight concentrations of the TiO_2 nanoparticles. Using a spectrophotometer the weight concentrations of the nanoparticles in the deionized water can be related to the light absorbance value [9] since the absorbance of a sample is proportional to the concentration of the absorbing species in the sample. In spectroscopy, the absorbance (also called optical density) is defined as [10]

$$A = \log(I_0/I) \quad (1)$$

where I and I_0 are the intensity of light that has passed through a sample (transmitted light intensity) and the intensity of the light before it enters the sample (incident light intensity), respectively. In this paper, the changes in the weight concentrations of the TiO_2 nanoparticles were estimated by the changes in the absorbencies, which were measured by a spectrophotometer (VIS-722S, Shanghai Lvyu Precision Instrument Manufacture Inc., China). Figs. 2 and 3 show the absorbencies A at wavelength 387 nm and the relative changes in A [i.e. $\Delta A = (A - A_0)/A_0$] of the rutile TiO_2 and anatase TiO_2 nanofluids versus time, respectively, where A_0 represents the initial absorbency when the preparation of the nanofluids were finished. For the 0.30 wt.% and 0.70 wt.% rutile TiO_2 and anatase TiO_2 nanofluids, ΔA are less than 2.4% within 286 h; while for the 0.05 wt.% rutile TiO_2 and anatase TiO_2 nanofluids, the absorbencies A decrease rapidly with time and ΔA are larger than 10.0% after 200 h. This implies that the 0.30 wt.% and 0.70 wt.% TiO_2 -water nanofluids kept stable for 286 h (about 12 days), but the stabilities of the 0.05 wt.% nanofluids were not so good.

2.2. DSC experiments

Crystallization analysis was performed using a DSC (204 F1 Phoenix, Netzsch, Germany) equipped with a cooler. The experiments were conducted under a nitrogen purge at flow rate of 20 mL/min. The temperature scale and the power response of the DSC were calibrated from the melting point ($156.61\text{ }^\circ\text{C}$) and the

enthalpy of fusion (28.47 kJ/kg) of 99.999% indium [11]. The TiO_2 -water nanofluids and deionized water were encapsulated in standard hermetically sealed aluminum crucibles. Matched empty crucibles were used as reference and baseline crucibles. The samples were first heated to $30\text{ }^\circ\text{C}$ and held in the state for 5 min to eliminate any influence of thermal history. Then the samples were cooled to $-30\text{ }^\circ\text{C}$ at $0.5\text{ }^\circ\text{C/min}$, $1.5\text{ }^\circ\text{C/min}$, $3.0\text{ }^\circ\text{C/min}$, $4.5\text{ }^\circ\text{C/min}$, $7.0\text{ }^\circ\text{C/min}$ and $9.0\text{ }^\circ\text{C/min}$, respectively. The DSC thermograms (exothermic curves) were recorded for data analysis.

From the DSC thermograms, the onset temperature $T_{c,onset}$ and the end temperature $T_{c,end}$ of crystallization can be obtained [12]. The latent heat of crystallization H_c can be calculated by integrating the exothermic curve from $T_{c,onset}$ to $T_{c,end}$ as [13]:

$$H_c = \int_{T_{c,onset}}^{T_{c,end}} (dH/dT)dT \quad (2)$$

where dH is the heat of crystallization in infinitesimal temperature difference dT .

The temperature T during non-isothermal crystallization can be transformed to crystallization time scale as:

$$t = (T_{c,onset} - T)/r \quad (3)$$

where r is the cooling rate. The half crystallization time $t_{1/2}$ is the time required for 50% crystallization to finish. The smaller the value of $t_{1/2}$, the faster the crystallization rate [14].

3. Results and discussion

The crystallization temperature T_c can be defined as the $T_{c,onset}$. The variation of the T_c of the TiO_2 -water nanofluids and deionized water are shown in Fig. 4. Obviously, at 0.5 – $9.0\text{ }^\circ\text{C/min}$, the shape of the T_c curve of the 0.05 wt.% rutile TiO_2 nanofluid was similar to those of the anatase TiO_2 nanofluids, like the symbol of inverse "N", indicating that the T_c decreased from $0.5\text{ }^\circ\text{C/min}$ to $1.5\text{ }^\circ\text{C/min}$, then increased from $1.5\text{ }^\circ\text{C/min}$ to $3.0\text{ }^\circ\text{C/min}$, and then decreased again from $3.0\text{ }^\circ\text{C/min}$ to $9.0\text{ }^\circ\text{C/min}$. Accordingly, the variation of T_c at 0.5 – $9.0\text{ }^\circ\text{C/min}$ could be divided into two regions by a certain cooling rate (named critical cooling rate). In the two regions, different nucleation mechanisms dominate. The first region is nucleation rate controlled region, where cooling rate is lower than the critical cooling rate and the T_c is determined by nucleation rate; while the second region is cooling rate controlled region, where cooling rate is higher than the critical cooling rate and the T_c is determined by cooling rate. For the 0.05 wt.% rutile TiO_2 nanofluid and the anatase TiO_2 nanofluids, the critical cooling rate could be $3.0\text{ }^\circ\text{C/min}$ according to the limited experimental data. In the nucleation rate controlled region, cooling rate is lower than the critical cooling rate; the cooling rate over the range from zero to the critical cooling rate is classified into low, middle, and high cooling rate in this paper. The cooling rate higher than the critical cooling rate is called

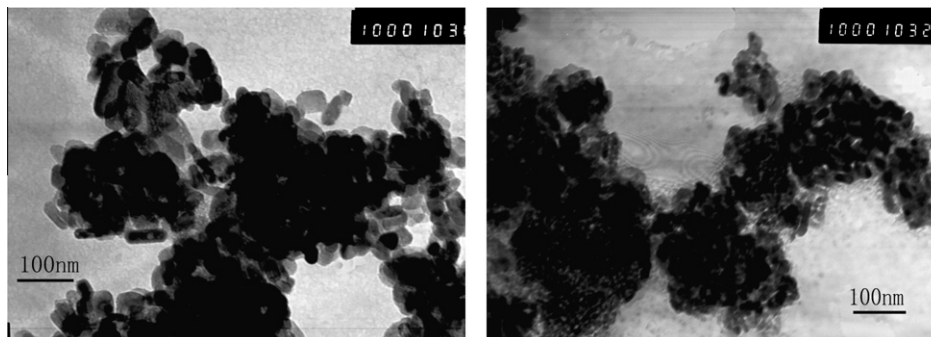


Fig. 1. TEM micrograph of the rutile TiO_2 nanofluid (left) and anatase TiO_2 nanofluid (right).

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