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# A novel method for determining the melting point, fusion latent heat, specific heat capacity and thermal conductivity of phase change materials

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ABSTRACT

In this paper, a novel method for determining the main thermophysical properties of phase change materials (PCM) is proposed, which can be called as "T-melting CHF" method. The melting point, fusion latent heat, thermal conductivity, and specific heat capacity in both solid and liquid phase of PCM, can all be obtained simultaneously by only one test. The theoretical fundamentals lying behind are introduced, the measurement apparatus and measurement principles are presented, and corresponding restrictive conditions for high accuracy measurement are provided. Theoretically, the thermophysical properties can be accurately measured by the proposed method if the test module can be perfectly insulated. However, heat loss inevitably exists in practical measurement, it will significantly influence the measuring accuracy, hence a correction method is proposed to improve this situation. A typical low melting point metal (gallium) and a typical organic PCM (n-eicosane) were tested to verify the feasibility and accuracy of the method. The experimental results agree well with the reference data reported in the literature. Compared with conventional measurement techniques, the method proposed here has advantages such as simple structure, low cost and high measurement speed; more importantly, it is able to measure multiple properties simultaneously.



## 1. Introduction

Phase change materials (PCM) are a class of materials which can absorb/release a large amount of latent heat during their melting/-solidification process, while their temperature remains nearly constant. PCM are widely used for thermal energy storage, they can meet the mismatch between energy supply and consumption [1]. For example, in concentrating solar power plants, intermittent solar energy can be partially stored by PCM for stable electric generation [2]; heat or cold can be generated and stored during midnight using the low-price off-peak electricity, and then be released for domestic or industrial usage in daytime [3,4]. PCM based thermal energy storage technologies have advantages such as high energy storage density, highly compactness, simple structure, and easy to maintain. Besides, PCM can also be used for thermal control of devices which work and generate heat intermittently, such as chips [5–8], photovoltaic cells [9–11] and power battery packs [12–14]. PCM can serve as a huge heat capacitor for thermal protection against high/low ambient temperature, which can be used for electronics in harsh environment [15], transportation of chemical or biomedical samples and foods [16]. Moreover, temperature conditioning of buildings can also be achieved by thermal buffering of PCM [17], which could greatly save their energy consumption.

Determination of the thermophysical properties of PCM is the basis for the researches and applications, the properties mainly include density, melting point, fusion latent heat, thermal conductivity, specific heat capacity, viscosity, thermal expansion coefficient, and so on. Generally, the phase change properties, such as melting point, fusion latent heat and specific heat capacity, can be measured by differential scanning calorimetry (DSC). The DSC method was proposed by Watson and O'Neill [18] in 1962, it has been developed to a sophisticated commercial thermal analysis technique. In DSC test, the temperature of the sample and the reference are designed to change linearly with time, the heat required to keep this change is measured as a function of temperature, and then the phase change properties can be determined.

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**Nomenclature**

| | |
|---|---|
| $A$ | coefficient in fitting curve (J/°C$^2$) |
| $A$ | sectional area (m$^2$) |
| $B$ | coefficient in fitting curve (J/°C) |
| $Bi$ | Biot number |
| $c_p$ | specific heat capacity (J/kg·K) |
| $D$ | diameter (m) |
| $Fo$ | Fourier number |
| $g$ | gravitational acceleration (m/s$^2$) |
| $\Delta H$ | fusion latent heat (kJ/kg) |
| $k$ | thermal conductivity (W/m·K) |
| $L$ | thickness (m) |
| $m$ | mass weight (kg) |
| $M$ | equivalent heat capacity (J/°C) |
| $q''$ | heat flux (W/m$^2$) |
| $q_{hs}$ | heating power of the heater (W) |
| $q_m$ | latent heat absorption (W) |
| $Ra$ | Rayleigh number |
| $R_0$ | electric resistance (Ω) |
| $s$ | solid-liquid interface location (m) |
| $\bar{s}$ | dimensionless form of $s$ |
| $Ste$ | Stefan number |
| $t$ | time (s) |
| $t_1$ | the time when the melting starts (s) |
| $t_2$ | the time when the melting ends (s) |
| $T$ | temperature (°C) |
| $T_0$ | ambient temperature (°C) |
| $T_m$ | melting point (°C) |
| $T_{mon}$ | monitoring temperature (°C) |
| $\Delta T_c$ | characteristic temperature difference (°C) |
| $V_0$ | voltage drop across $R_0$ (V) |
| $V_1$ | voltage drop across the heater (V) |
| $W$ | width of PCM slab (m) |
| $x$ | rectangular coordinate (m) |
| $\bar{x}$ | dimensionless form of $x$ |

*Greek symbols*

| | |
|---|---|
| $\beta$ | thermal expansion coefficient (1/K) |
| $\theta$ | relative temperature to $T_0$ (°C) |
| $\Theta$ | dimensionless temperature |
| $\mu$ | viscosity (kg/m·s) |
| $\rho$ | density (kg/m$^3$) |

*Abbreviations*

| | |
|---|---|
| *CHF* | constant heat flux |
| *DSC* | differential scanning calorimetry |
| *PCM* | phase change material |
| *PTFE* | poly tetra fluoroethylene |

*Subscript*

| | |
|---|---|
| *af-m* | after-melting process |
| *ave* | average value |
| *Al* | Al plate |
| *be-m* | before-melting process |
| *c* | critical value |
| *du-m* | during-melting process |
| *hs* | heat source (heater) |
| *ini* | initial value |
| *l* | liquid phase |
| *loss* | heat loss |
| *no-load* | no-load test |
| *refl* | reflection point |
| *s* | solid phase |
| *w* | wall, upper surface of Al plate |

Although the DSC method has been widely recognized and used, there still exist some shortcomings: (1) The instrument is somewhat complex and expensive, and the test is costly; (2) Only a very small amount of sample can be used in the test for accuracy purpose, say 1–30 mg (about several mm$^3$); however, in practice, bulk PCM are usually used (several cm$^3$, even to several m$^3$), the properties of the very small sample of PCM might be different from the bulk one, especially for these with heterogeneous additives [19]. Besides, for some hybrid PCM which are mixture of different bulk materials, their equivalent macroscopic thermophysical properties are hard to be determined by the small sample. Hence, it is of practical significance to develop a simpler method for the measurement of phase change properties, which would be much helpful for reducing the test cost, especially for situations where there exists large amount of samples. In addition, the method should be capable of measuring the properties of bulk samples.

In 1999, Zhang and Jiang [20] proposed a T-history method, which is able to measure the fusion latent heat, specific heat capacity and thermal conductivity of several bulk samples of PCM simultaneously. The T-history method monitors the temperature curves of the PCM sample and a well-known reference sample (e.g. water) during their natural cooling process, lumped capacity method is used to analyze the process and then corresponding thermophysical properties can be determined. Compared with conventional DSC method, the T-history method greatly simplifies the test instrument, and thus the cost can be greatly reduced. In the pioneering work by Zhang and Jiang [20], the relative deviations between the test results obtained by the T-history method and these reported in the literature are less than 10%, which can satisfy the requirement of engineering applications.

The T-history method is widely accepted once after it was proposed, and many efforts have been done to further improve this technique. In 2003, Hong et al. [19,21] improved the accuracy of the T-history method by correcting some improper assumptions in the original model, such as using a degree of supercooling as the end of latent heat period and neglecting sensible heat during phase change. Marín [22] considered the temperature dependence of the properties and built an enthalpy conservation equation in small time interval to describe the natural cooling process of the samples, and thus the enthalpy-temperature curve can be obtained. In 2006, Peck et al. [23] found that for PCM whose melting point is lower than the ambient temperature, the temperature gradient of the PCM in the vertical direction is significant and might lead to inaccuracy of the measurement of fusion latent heat, it was proposed that setting the test tube horizontally could improve this situation.

The common features of the two methods mentioned above are that: (1) a well-known standard reference sample is needed; (2) lumped capacity method is used to model the thermal process. To satisfy the perquisite of the lumped capacity method, the temperature inside the PCM sample should be uniform enough. Hence, in DSC test, very small amount of sample is used and low heating/cooling rate is applied, which makes it unable to measure bulk samples. In T-history method, low Biot number $Bi$ must be satisfied ($Bi < 0.1$), hence the natural cooling process is adopted, this makes the test somewhat time-consuming, generally about 1 h is needed; it even takes tens of hours in order to get higher accuracy [24]. Besides, in DSC test, the thermal conductivity of the PCM sample cannot be obtained; in T-history method, an extra test based on another principle is needed in order to get the thermal conductivity.