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# TRANSMISSION CHARACTERISTICS OF ULTRASONIC WAVES IN FINE PARTICLES OF PHASE-CHANGE MATERIAL-WATER SLURRY

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#### ABSTRACT

Phase Change Material Slurry (PCMS) consists of multi-component and multi-phase suspensions. The micro emulsion phase change material slurry (MEPCMS) is composed of paraffin as a phase change material, water as a carrier fluid, and surfactant as an emulsifier. This type of PCMS is known as a candidate of functional thermal fluids, which show very high energy density, and allow to save pumping energy [1]. Sound velocity in a slurry layer is an important factor for measuring velocity profile of the slurry layer with a Ultrasonic poppler Method (UDM) [2] or to characterize the phase change in multi-phase media [3], [4]. A sound speed measurement device is selected in the present study of avoiding the parameter of the geometrical dilatation when temperature change occurs. The principle of measurements is based on the differential time of ultrasonic wave flight. The present measuring device works for sound speed of a distilled water, and it is now tested to measure the flow velocity of the MEPCMS.

#### INTRODUCTION

Phase Change Material Slurries (PCMS) are composed of multi component and multi-phase suspensions. These PCMS as functional thermal fluids are researched and developed, which show very high energy density and high pumping energy saving. Interesting flow behavior has been observed with this type of PCMS, however the opacity of this type of PCMS disturbs to visualize the flow pattern. In order to use a Ultrasonic Doppler Method (UDM) [2] for measuring velocity of PCMS. it is important to obtain an accurate sound speed in a PCMS layer.

Often the sound speed is experimentally determined by a method of the Time Of Flight (TOF). Sound speed measurement systems available on the market are based on this principle. This principle is sensible on the geometrical dilatation when temperature change occurs. The sound speed measurement is useful to specify a substance. The settlement of phase state of a PCM in a PCMS can be made by a DTF [4].

## COMPOSITION OF THE MICRO-EMUSLION

The micro-emulsion phase change material slurry (MEPCMS) used in the present experiment is composed of 30 % (mass percent) of paraffin, 65 % of water and 5 % of surfactant. This PCMS is well known as a candidate of functional thermal fluids, which shows very high energy density and low pumping energy per transmitted heat [1]. Fine parraffin particles are distributed in its diameter range from 0.1 μm to 1.2 μm

(its average diameter of 0.51  $\mu$ m). The particle-frequency shows a lognormal function against particle diameter [5]. The PCM is solid when the temperature of MEPCMS is below its melting point of 47°C. Above this temperature, the paraffin melts and the MEPCMS is totally in a liquid condition. The PCM in the present study is composed of several kinds of n-paraffin ( $C_nH_{2n+2}$ ). Its major component of the PCM is tricosane  $C_{23}H_{48}$ .

The appearance of the MEPCMS has a milk-like white color. The polymer surfactant having hydrophilic radicals in the water side and hydrophobic radicals in the paraffin side, acts as an emulsifier or a covering film separating the PCM from water. The PCM particles and water are immiscible each other [5].

The density of the paraffin has significant temperature dependence. Generally the density of paraffin with linear chain structure depends on the previous history of pressure and temperature [6]. Density measurement of MEPCMS used in the present experiment was done in the reference [5], as shown in Fig.1. It is show in the literature [4], [5] that the density and the volume of the dispersed phase exert a strong influence on the sound speed. The morphology of the PCM as a dispersed phase is some complicated, but the present result does not consider its complexity like shape and size distribution of the PCM particles.

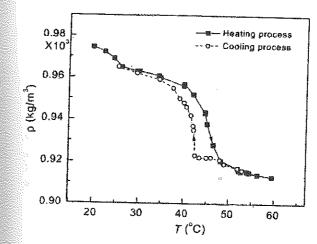


Figure 1. Density measurement of the micro emulsion, measurement from [5].

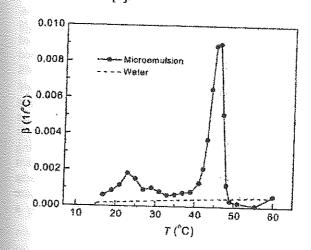


Figure 2. Volume expansion coefficient against temperature for the micro emulsion, measurements from [5].

It is seen in the Fig. 2 that there are two local maximum volumetric expansion coefficients in the temperare range from 15°C to 60°C. One is at temperature of about 23°C (crystal structure transition temperature) and the other is at temperature of about 47°C (the melting point). The same peak appears in the measurement of the apparent specific heat capacity [5].

## SOUND SPEED MEASUREMENT DEVICE USING THE DIFFERENTIAL TIME OF FLIGHT (DTF)

To avoid measuring error due to the geometrical variation when the phase change of the PCM occurs, the principle of sound speed measurement in the present study is based on the Differential Time of Elight (DTF) [8]. The measurement of the sound speed is performed in multiple steps. An ultrasonic transducer is placed at a known distance from a reflecting wall. The transducer emits a burst of beam, which is reflected by the wall and returns back to the transducer. The time of the reflected echo after the emission (dt) is recorded on an oscilloscope. After the first measurement, we shift the position of the transducer with a micrometric screw

(dx). The difference of the TOF between two measurements corresponds to two times the displacement of the transducer. The present measurement repeats this operation several times and obtains dt-dx curve. The sound speed is given by the slope of this curve. The slope and its accuracy are determined by a classic least square method, as given in [7].

The container packed with the MEPCMS is composed of a plexiglass cylinder with a volume of about two liters. A micrometric screw is mounted on the top of its container to control and measure the dispacement of the ultrasonic transducer. The reflecting wall is in stainless steel sheet. The height from the transducer and the reflected wall is around 7 cm. A mixing element with two proppelers is located inside the container with the aim to avoid stratification of the sample. The prototype offers the possibility to test different frequency of US beam.

The validation of the present measuring method was confirmed using a distilled water. The ultrasonic signal, emit by a function generator device Tabor 8550, is a burst of 2 periods with an amplitude of 16 V peak to peak. The frequency of repetition is adjustable. The echo signal was observed by an Iwatsu DS-8814 digital oscilloscope. The temperature is measured with a thermocouple with an accuracy of 0.1 K. Measurements are made at different temperature and the obtained dara is compared with values obtained by Grosso and Mader modified by Povey [4]. Results are shown in Fig. 3. Systematic errors seem to occur for the performed validation's measures at three frequency, 2and 8 MHz. In the case of 4 MHz, measuring results of the sound speed show a lower random error. At this stage of the developement of the DTF, the frequency of 4 MHz seems to be most appropriate for the measurement.

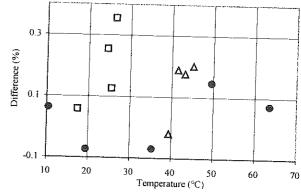


Figure 3. Results of the validation of the sound speed by the prototype based on the principle of the differential time of flight (DTF). The difference in % between the approximation equation of del Grosso and Mader modified by Povey [4], and measurement performed by DTF is shown by the graphic. Medium of measurements is distilled water. Squares represent results for a signal of 8 MHz, black rounds for 4 MHz and triangles 2 MHz.

### MICRO-EMULSION SLURRY SOUND SPEED

The US signal attenuation of the MEPCMS layer is too strong to use the same signal (4 MHz - 16 V peak to peak) in the temperature range in the present measurement. Because of the parrafin particle diameter, scattering of the beam is more probable than absorption of the signal by the PCM particle. It is the reason why we finally choose the shift of the frequency of the US signal, from 4 to 2 MHz for measuring the MEPCMS. The penetration of the beam is stronger when reducing the frequency. The chosen amplitude for the US beam is 150 V. The signal is control and emit by a UVP instrument. One series of measurements was performed with an increase in the temperature of the MEPCMS, starting from the ambient temperature. The other series was done during the cooling process, starting with a temperature above the solidification point of the PCM. Every measurement was done after a stabilization of the temperature. Results of these measurements are shown in the Fig. 4.

Because the container is not hermetic, some evaporation of water can occur. In the present experiment, the water content in the MEPCMS can decrease during the measurement. The water content in the sample was 57 % at the end of the measurement.

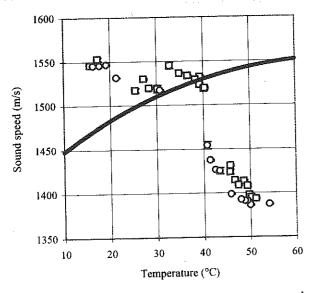


Figure 4. Results of the sound speed measurements by DTF method on a micro-emulsion slurry. Square points show measurements obtain with the increase of the temperature sample between successif point. Circle point show measurements obtain at stabilizate temperature, with cooling between two of them. The black line represents sound speed in water, from [4].

Regarding the Fig. 4, a dramatic variation in the sound speed appears in the case of the MEPCMS; whether the PCM particle are liquid or solid. This behavior is similar to the density curve of the MEPCMS – see Fig.1. Hysteresis on sound speed against temperature appears for the heating and the cooling process. In the reference [8], the authors have reported about a hysteresis about

10 K for most paraffins, wich is higher than 1 K reprorted for mono paraffin. This phenomenon appears also in the reference [4] with other PCMs when the sound speed was measured.

After multiple-use of the same MEPCMS, some problems appeared. Measurements were not possible anymore. The solidification of the parrafin was not in micro particles, but in one large block, in which MEPCMS particles agglomerated each other. That is, some doubt appears of variation in shape and size of the MEPCMS during solidification of the PCM After trying to dissolve the parrafin by melting, stratification of materials appeared. After mixing and cooling the MEPCMS, same problem of agglomeration remained.

#### CONCLUSION

A device to measure sound speed in the MEPCMS was developed with the aim to avoid measuring error of the volumetric variation when temperature change occurs. The method is based on the Differential Time of Flight DTF. Measurement in a distilled water showed reasonable results with a high accuracy. This method was applied to measure the sound speed of a MEPCMS at various temperatures. Effort must be done to avoid water evaporation from the MEPCMS during measurements. Some doubt appears concerning the capacity of the MEPCMS to take the same form when it is cooled down from a temperature above the melting point. Sound speed in MEPCMS is an important quantity for many reasons, for example, to characterize the phase state in multi-phase media, to measure accurate velocity profile in the MEPCM layer by a Ultrasonic Doppler Method.

#### **ACKNOWLEDGEMENT**

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THERMAL CONDUCTIVITY OF EMULSION AS FUNCTIONAL FLUID FOR ICE STORAGE
(Discussion on Temperature Dependence of Emulsion)

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One of authors had studied the functional fluid as the thermal storage material for ice storage. The functional fluid was cooled with stirring, and then it changed into ice slurry with good fluidity. The functional fluid developed had many excellent features when it was cooled. However, it had one weak point that a latent heat drops with freezing. Hence, two kinds of emulsions were developed as a new functional fluid. On the basis of discussion on structure of emulsions, it was found that one was W/O type emulsion and the other was O/W type emulsion. Then, validity of those emulsions for ice storage is discussed now.

In this paper, we report the experimental results on thermal conductivities of those emulsions which are important thermal property. We used a transient hot wire method to measure thermal conductivity. Experiments were carried out under the condition of 0,5,10,15 and 20℃. Measurements over 20 times were repeated for one fixed temperature. We adopted the average value of many measurements as the experimental result. Before thermal conductivities of those emulsions were measured, thermal conductivity of distilled water was measured to examine the experimental apparatus. From comparison of the experimental result for thermal conductivity of distilled water with that for the reference value, good agreement between both was obtained. Two kinds of emulsions were made of water-silicone oil mixture with a little modified silicon oil with amino group having hydrophobic property. The compositions of two emulsions were water of 89.2vol%(495 ml), silicone-oil of 9.9vol%(55ml) and modified silicon oil with amino group of 0.9vol%(5ml), respectively. The ratio of water to silicone oil was 9:1. From experiments, It was found that thermal conductivities of two emulsions increased linearly with increase of temperature, respectively. Therefore, thermal conductivities for two kinds of emulsions were approximated as straight line by the least square method, respectively. Moreover, the experimental result of thermal conductivity for W/O emulsion was less than that for O/W emulsion by about 5.3% on average in spite of the same composition. The experimental results for O/W and W/O emulsions were compared with calculated values obtained by Maxwell equation, good agreements between experimental results and calculated values were obtained, respectively. At last, inaccuracy of measurement was discussed, it was  $\pm 2.29\%$  for O/W emulsion, while, it was  $\pm 1.99\%$  for W/O.

#### 1. 緒言

ダイナミック型氷蓄熱システムは熱負荷追従性に優れ、冷熱の運搬も容易であり、優れた氷蓄熱システムである。本研究室ではダイナミック型氷蓄熱システムの新たな畜熱材として、水とシリコンオイルの混合液に微量の疎水性のアミノ変性オイルを加えたものを攪拌によって乳化きせ、O/W 型エマルション及び W/O 型エマルションを生成する。そしてそれらを攪拌・冷却させながら、サスペンションを生成する研究を行っている。

熱伝導率は研究・開発を行うのにあたって大変重要な物性値ではあるが、今回生成された2種類のエマルションの信頼すべきデータは未だない。そこで、本研究ではのW型エマルション及びW/O型エマルションの熱伝導率を非定常線熱源法により測定した。そしてそれぞれのエマルションの温度を変えながら測定を行い、得られた結果に対して検討・考察をした。

#### 2. 実験

2-1. エマルションの生成条件と生成方法 本研究で生成した O/W 型エマルション及びW/O 型エマ Table 1 Composition of emulsion

Table 1 Composition of emulsion	
Silicone oil (9.32×10 <sup>-3</sup> [Pa·s])	55ml
Tap water	495ml
Amino group modified silicone oil	5ml
Ratio of water to oil	9:1

ルションの組成は水 495ml、シリコンオイル 55ml、添加剤としてアミノ変性オイル 5ml である。その材料及び組成比を Table1 に示す。水と油の体積比は 9:1 とした。

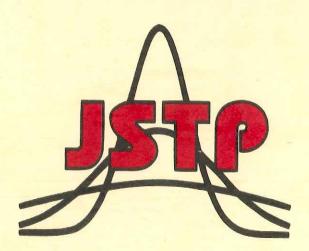
O/W 型エマルションは、Figure1 に示す実験装置を用いて生成される。所定のシリコンオイル、アミノ変性オイル、水道水をビーカーの中に入れ、超音波発生装置を使用してこの混合液に 420W 相当の超音波を 3 分間照射する。混合液は、超音波によってキャビテーションが起こり攪拌され、O/W型エマルションが生成される。

W/O 型エマルションは、Figure2 に示す実験装置を用いて生成される。所定のシリコンオイルとアミノ変性オイルをビーカーに入れ、それを 300rpm で攪拌しながら水道水を入れる。水道水を入れ終わったらそのまま

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