

# Development of Laser-Induced Capillary Wave Method for Viscosity Measurement Using Pulsed Carbon Dioxide Laser<sup>1</sup>

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A new experimental apparatus, based on the laser-induced capillary wave method involving the use of a pulsed carbon dioxide laser (wavelength 10.6  $\mu\text{m}$ , pulse width 50 ns, power 65 mJ) as a heating source has been developed. Since the present technique is applicable to a wide range of viscosity, this method is applicable to the process in which the viscosity drastically changes within a short period of time. In this method, interfering laser beams heat a liquid surface and generate a capillary wave (the wavelength can be adjusted from 20 to 200  $\mu\text{m}$ ) caused by a spatially sinusoidal temperature distribution. The temporal behavior of the capillary wave is detected by a diffracted probe beam (He-Ne laser, 15 mW) at the heated area. The dynamics of the capillary wave provide information regarding thermophysical properties such as viscosity and surface tension. In the present study, several liquid samples spanning the viscosity range from 0.33 to 7080 MPa·s (e.g., acetone, toluene, 1-hexanol, ethylene glycol, JS1000, and JS14000) were investigated at room temperature. The detected signals for several liquid samples exhibiting a wide range of viscosity agree well with theoretical calculations, taking into account the influence of the distribution of surface tension.

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**KEY WORDS:** laser-induced capillary wave; measurement technique; surface tension; viscosity.

## 1. INTRODUCTION

Reliable information regarding the viscosity of liquids is essential in many fields of science and industry where it is important to control and design

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<sup>1</sup>Paper presented at the Fifteenth Symposium on Thermophysical Properties, June 22–27, 2003, Boulder, Colorado, U.S.A.

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liquid flow. The viscosity of fluids has been successfully measured by diverse techniques such as the capillary viscometer, the oscillating-body viscometer, the vibrating viscometer, and the falling-body viscometer [1, 2], all of which have been well established as standard viscosity measurement techniques. Users of viscometers choose each of these techniques depending on their sample viscosity range and required uncertainty. Although the above-mentioned conventional viscosity measurement techniques are very useful to determine a limited range of liquid viscosity with high accuracy, a non-contact measurement technique applicable to the process in which the viscosity drastically changes within a short period of time has not been available so far. There are many examples of such processes in a wide variety of industries. For instance, in a food processing plant, the viscosity of liquid foods changes considerably during processing, and determination of the viscosity is essential in the design and control of such processes and is also useful for the evaluation of food quality [3]. Therefore, a new non-contact technique is needed that can be used to measure significantly changing viscosities by means of a single apparatus. In the present study, we have developed a new apparatus to detect a laser-induced capillary wave generated by the interference of pulsed carbon dioxide laser beams.

A number of studies have focused on non-contact techniques to measure viscosity and surface tension by detecting the dynamics of capillary waves existing on a liquid surface. For example, Rhim et al. [4] measured the surface tension and the viscosity of molten zirconium up to about 2200 K by detecting the characteristic oscillation frequency and the decay time of a liquid zirconium drop which was electrostatically levitated. The drop oscillation was externally induced by superimposing a small sinusoidal electric field on the levitation field. Another example of a non-contact technique to measure a liquid surface tension and viscosity can be performed by observing the spontaneous capillary wave (rippion) caused by thermal fluctuations. Since this surface laser-light scattering technique (SLLS) was initiated by Katyl and Ingard [5, 6], the technique has been applied to a wide variety of liquids such as polymer solutions, liquid crystals [7], high temperature molten silicon, and lithium niobate [8–10], as well as to the accurate measurement of toluene [11]. However, when we apply the SLLS technique to measure highly viscous liquids such as glycerol and silicon oils, the spontaneous capillary waves are completely over-damped. For such high viscosity liquid samples, the experimental determination of viscosity by SLLS becomes very difficult because the frequency spectrum width turns out to be less than one hertz. Consequently, a very long time (about 30 min) is required to take a single viscosity measurement. In such a low frequency region, various noise sources may overlap with the weak signal from the capillary wave [12, 13].

In order to overcome the shortcomings of the SLLS technique for highly viscous liquids, it may be possible to develop a new principle of viscosity measurement by replacing the weak and random fluctuations (spontaneous capillary wave, Brillouin scattering) by strong and coherent excitations from a laser-induced grating (laser-induced capillary wave, forced Brillouin scattering). This idea is analogous to the development of forced Rayleigh scattering from spontaneous Rayleigh scattering for measurement of thermal diffusivity [14]. The first forced Brillouin scattering experiment was performed by Terazima's group [15, 16] in 1999. They generated a capillary wave by crossing two excitation beams of the third harmonics of a Nd: YAG laser, and observed a strong over-damped wave from the air/hexanol surface at a large wave number. Subsequently, Terazima's group revealed the mechanism of the laser-induced capillary wave creation by solving the phenomenological equations, and compared the observed signals with the theoretically calculated waveforms [13]. In the meantime, the author's group independently started to develop a new contact-free measurement technique to determine a wide range of viscosity by observing the laser induced-capillary wave generated by the interference of a pulsed carbon dioxide laser [17, 18]. By employing an infrared laser beam to excite the IR absorption band of liquid samples, the capillary wave can be induced without adding any light absorbing material (dye) to "pure" liquids. Therefore, heating by a carbon dioxide laser has an advantage over heating by a Nd: YAG laser, especially in the case when the technique is applied to *in situ* determination of the viscosity of liquid foods or bio-fluids into which the addition of impurities is obviously prohibited.

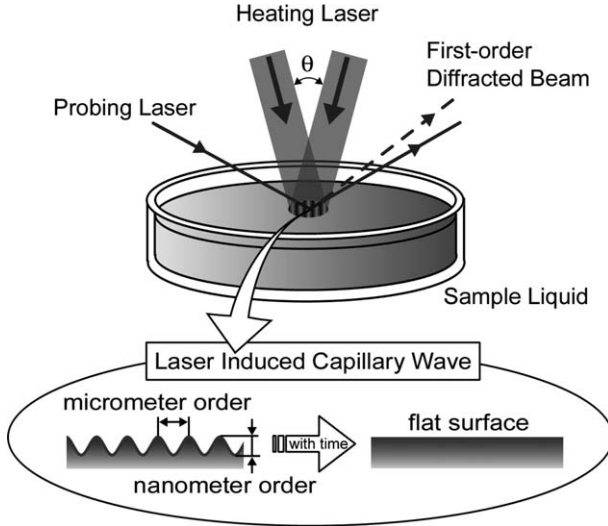
In the present paper, we describe the principle of measurement and the experimental apparatus when using a pulsed carbon dioxide laser, and discuss comparisons of the detected signals with theoretical calculations for several liquid samples exhibiting a wide range of viscosities.

## 2. PRINCIPLE OF MEASUREMENT

### 2.1. Creation and Detection of Laser-Induced Capillary Wave

The principle of the laser-induced capillary wave technique is shown schematically in Fig. 1. In this method, pulsed high-power laser beams of equal wavelength and equal intensity intersect on a sample surface under a crossing angle  $\theta$ . They generate an optical interference fringe pattern as illustrated in Fig. 1, whose intensity distribution is spatially sinusoidal. The laser intensity distribution and wave number can be written as

$$I(x) = I_h \{1 + \cos(kx)\}, \quad (1)$$



**Fig. 1.** Principle of the laser-induced capillary wave method. The laser-induced capillary wave has micrometer-order wavelength and nanometer-order amplitude.

$$k = \frac{2\pi}{\Lambda} = \frac{4\pi \sin(\theta/2)}{\lambda_h}, \quad (2)$$

where  $I_h$  is the heating laser beam intensity,  $\Lambda$  is the fringe spacing, and  $\lambda_h$  is the wavelength of the heating laser. At the end of heating, a corresponding spatially sinusoidal temperature distribution induced by the grating pattern is

$$T(x) = T_m + \Delta T \cos(kx), \quad (3)$$

where  $T_m$  is the mean initial temperature rise and  $\Delta T$  is the amplitude of the spatially periodic temperature distribution.

The temperature distribution of the thermal grating creates a capillary wave on the liquid surface driven by the thermal expansion and the temperature dependence of the surface tension. The displacement of the capillary wave along the  $z$  direction can be expressed by

$$u_z = u_m + \Delta u \cos(kx), \quad (4)$$

where  $u_m$  is the spatially uniform displacement and  $\Delta u$  is the amplitude of the laser-induced capillary wave. In the present experimental setup, the amplitude of the created capillary wave is estimated to be less than 10 nm.

When the probing laser beam is incident on the heating area, it is diffracted because the capillary wave acts like a reflection grating. The first-order diffraction efficiency is directly proportional to the square of the surface displacement as shown in the following equation:

$$I(t) \propto \Delta u(t)^2, \quad (5)$$

where  $I(t)$  is the intensity of the first-order diffracted beam. Therefore, we are able to obtain information regarding the behavior of the laser-induced capillary wave by measuring the time profile of the first-order diffracted beam. Particularly, the temporal behavior of the laser-induced capillary wave is described in Sections 2.2. and 2.3. Also, in the present technique we employ a short-pulsed CO<sub>2</sub> laser in order to disregard the complex motion of the laser-induced capillary wave in the course of the heating process.

## 2.2. Motion of Laser-Induced Capillary Wave

The motion of the laser-induced capillary wave is described by three fundamental phenomenological equations under several boundary conditions. The displacement of the Newtonian liquid surface has been reported by Yasumoto et al. [13] and is summarized as follows.

First, the Navier–Stokes equation is

$$\frac{\partial^2 \mathbf{u}}{\partial t^2} - \nu \nabla^2 \frac{\partial \mathbf{u}}{\partial t} - V_L \nabla \nabla \cdot \mathbf{u} = -\beta V_L^2 \nabla T, \quad (6)$$

where  $\mathbf{u}$  is the displacement,  $\beta$  the thermal expansion coefficient,  $V_L$  the sound speed in the liquid, and  $\nu = \eta/\rho$  is the kinetic viscosity ( $\eta$ : the viscosity,  $\rho$ : the density of the medium). Here, in order to justify the assumption that the laser-induced capillary wave is similar to the spontaneous capillary wave, we assume that the displacement of the capillary wave on the surface is small relative to the wavelength. In addition, the convective term is neglected. Second, the continuity equation is expressed as

$$\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \mathbf{V}) = 0, \quad (7)$$

where  $\mathbf{V}$  is the velocity of the liquid. Third, the heat conduction equation can be obtained by the following equation:

$$\rho C_p \left( \frac{\partial T}{\partial t} \right) - \lambda \nabla^2 T = I_h \alpha [1 + \cos(kx)] \exp(\alpha z) \delta(t), \quad (8)$$

where  $\lambda$  is the thermal conductivity,  $\alpha^{-1}$  is the optical absorption length of the heating beam,  $C_p$  is the isobaric heat capacity, and  $\delta(t)$  is the Dirac delta function.

The boundary conditions are as follows: the velocity of the liquid should be zero sufficiently far from the surface, and the thermal conduction from the liquid to the gas phase is neglected. In addition, the tangential and normal components of the stress to the surface, assumed under the conditions that the stress acting from within the liquid is exactly counterbalanced by the stress due to the surface force since no stress can exist in the gas phase, is given by

$$\sigma \frac{\partial u_x}{\partial x^2} - 2 \frac{\partial}{\partial t} \left( \frac{\partial u_z}{\partial z} \right) + \rho V_L^2 \left( \alpha T - \frac{\partial u_x}{\partial x} - \frac{\partial u_z}{\partial z} \right) = 0, \quad (9)$$

$$\eta \frac{\partial}{\partial t} \left( \frac{\partial u_x}{\partial z} + \frac{\partial u_z}{\partial x} \right) - \left( \frac{\partial \sigma}{\partial T} \right) \frac{\partial T}{\partial x} = 0. \quad (10)$$

Especially, the temperature dependence of the surface tension is considered by using Eq. (10) in which the surface-tension distribution term is included.

The amplitude of the laser-induced capillary wave can be obtained by solving Eqs. (4) and (6)–(10) using a Laplace transformation. The Laplace transformed amplitude of the wave  $\Delta U$  can be written as [19]

$$\begin{aligned} \Delta U = & \frac{\alpha \beta I_e}{\lambda k^2} \frac{b_1}{(b_2^2 - b_1^2)} \\ & \times \left[ \frac{b_1 b_5}{1 + b_5} \left\{ -\frac{e^{b_1 k z}}{b_4^2 - b_1^2} + \frac{e^{b_2 k z}}{b_4^2 - b_2^2} \right\} - \frac{b_2 - b_1}{b_2} \left( C_1 e^{b_3 k z} + b_4 C_2 e^{b_4 k z} \right) \right], \end{aligned} \quad (11)$$

where

$$b_1 = \alpha / k, \quad (12)$$

$$b_2 = \left( 1 + s / \alpha k^2 \right)^{1/2}, \quad (13)$$

$$b_3 = \left( 1 + s / \nu k^2 \right)^{1/2}, \quad (14)$$

$$b_4 = \left[ 1 + \frac{s^2}{(V_L^2 + s \nu) k^2} \right], \quad (15)$$

$$b_5 = \frac{V_L^2}{s \nu}, \quad (16)$$

and both  $C_1$  and  $C_2$  are extracted by solving the determinant [19]

$$\begin{aligned} & \begin{bmatrix} 2b_3 + d_1b_5 & 2b_4^2 + b_5(b_4^2 + d_1b_4 - 1) \\ 1 + b_3^2 & 2b_4 \end{bmatrix} \begin{bmatrix} C_1 \\ C_2 \end{bmatrix} \\ & = \begin{bmatrix} d_2d_3 - b_5 \\ 2b_1b_2(b_1 + b_2)d_3 - \frac{\partial\sigma}{\partial T} \frac{k}{\beta\rho V_L^2} b_5 \end{bmatrix}. \end{aligned} \quad (17)$$

Here [19]

$$d_1 = \frac{\sigma k}{\rho V_L^2}, \quad (18)$$

$$d_2 = b_1b_2 \left\{ (b_5 + 2) (b_4^2 + b_1b_2) + d_1b_5 (b_1 + b_2) \right\} - b_5 (b_1^2 + b_1b_2 + b_2^2 - b_4^2), \quad (19)$$

$$d_3 = \frac{b_5}{(1 + b_5) (b_4^2 - b_1^2) (b_4^2 - b_2^2)}. \quad (20)$$

The time profile of the liquid motion is numerically calculated from Eq. (11) by using the fast inverse Laplace-transform algorithm [20].

### 2.3. Mechanism of Laser-Induced Capillary Wave Generation

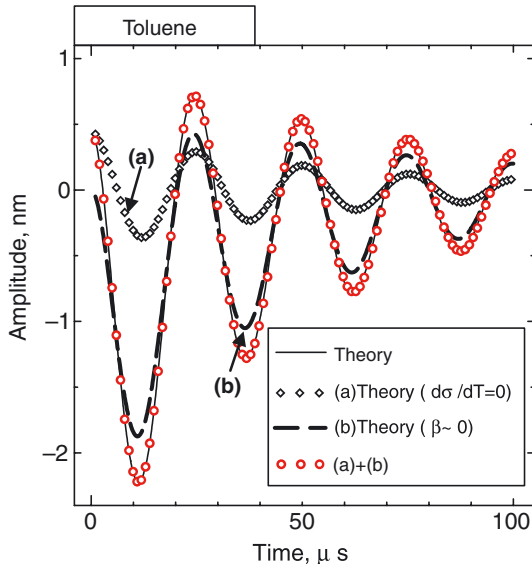
In the early stages of our study, we assumed that the motion of a laser-induced capillary wave is similar to that of ripples [21, 22]. However, as pointed out by Terazima's group [13], we have found that the influence of the temperature dependence of the surface tension is extremely significant under the present experimental conditions. Since the laser-induced capillary wave is generated by the temperature distribution, there is a resulting surface tension distribution. That is to say, it is necessary to consider the influence of the temperature dependence of the surface tension, and we thus apply Eq. (10) to one of the boundary conditions. The temperature dependence of the surface tension is one of the important factors involved in the behavior of laser-induced capillary waves.

Figure 2 shows the amplitude of the laser-induced capillary wave as determined by Eq. (11) using the fast inverse Laplace transform on a surface of toluene as an illustration of a low viscosity liquid. The thermo-physical properties of toluene which we have used in these calculations are listed in Table I. Wave (a) is calculated under the assumption that the surface tension is not temperature dependent. In the calculation of (b), it is assumed that the thermal expansion is negligible. The sum of (a) and (b) is equal to the theoretical amplitude of the laser-induced capillary wave,

and (b) is the dominant motion of the laser-induced capillary wave, i.e., the temperature dependence of the surface tension is one of the significant factors for the wave generation. Accordingly, we have demonstrated that, under the present experimental conditions, the temperature dependence of the surface tension, as well as thermal expansion, is an essential contributor to wave generation. However, for a high viscosity liquid the dominant contribution for the wave generation is not the temperature dependence of the surface tension but thermal expansion.

### 3. EXPERIMENTAL APPARATUS

Figure 3 shows the present experimental apparatus. A pulsed CO<sub>2</sub> laser (Edinburgh Instruments Ltd., MTL-3; wavelength 10.6  $\mu\text{m}$ , pulse width 50 ns, output energy 65 mJ) is employed as a heating source. Repetition rates are available for a single shot to 100 Hz. Especially, because of the short-pulsed laser beam, it is possible to neglect the complex behavior of the laser-induced capillary wave in the course of the heating process. The pulsed heating beam is positioned vertically up mirror 1 (M1) and divided into two beams of equal intensity by means of a beam splitter (BS). They intersect on the sample surface by reflecting on M2, M3, and



**Fig. 2.** Theoretical calculation of the amplitude of a laser-induced capillary wave versus time for toluene.



**Table I.** Thermophysical Properties of Toluene Used in Theoretical Calculation (at 298 K and 1 atm.) [23]

Viscosity ( $\eta$ )	0.552 mPa·s
Surface tension ( $\sigma$ )	27.3 mN·m <sup>-1</sup>
Temperature dependence of surface tension ( $d\sigma/dT$ )	-0.118 mN·m <sup>-1</sup> ·K <sup>-1</sup> (283–373 K) [24]
Density ( $\rho$ )	862 kg·m <sup>-3</sup>
Thermal expansion ( $\beta$ )	1060×10 <sup>-6</sup> ·K <sup>-1</sup> (273–303 K)
Thermal conductivity ( $\lambda$ )	133 mW·m <sup>-1</sup> ·K <sup>-1</sup>
Heat capacity ( $C_p$ )	1.717 kJ·kg <sup>-1</sup> ·K <sup>-1</sup>
Optical absorption length ( $\alpha^{-1}$ )	5.9 <sup>-1</sup> m (@ 10.6 $\mu$ m) [25]
Sound speed ( $V_L$ )	1130 m·s <sup>-1</sup>

M4 to produce an interference pattern. These optical devices are fixed on a vertical optical bench, and the radius of the heating beams is approximately 6 mm on the sample surface. Typically the range of fringe spacing of the grating is adjusted from about 20 to 200  $\mu$ m. The tunable frequency range of the capillary wave corresponding to the adjustable fringe spacing of the present apparatus is from about 5 kHz to about 1 MHz. The liquid sample is filled in a petri dish (depth 1.6 mm, radius 5 cm). The probing laser is a He–Ne laser (wavelength 632.8 nm, output power 15 mW, beam radius 2 mm). The diffracted light signal as a function of time is detected by a photomultiplier tube (PMT; Hamamatsu, R928) through a pinhole (PH) and an interference filter (IF). The output signal is sent to a digital storage oscilloscope (DSO) and is transferred to a computer. The entire apparatus is arranged on an optical bench.

In order to determine the fringe spacing prior to the experiment, the CO<sub>2</sub> laser beams intersect on the surface of the transparent PMMA plate (thickness 2 mm), which is set at the same position as the sample surface. Consequently, the grating pattern that is generated by the same optical configuration as the experiment is printed on the surface of the PMMA plate. When the He–Ne laser is incident on the printed grating, the diffracted beam is generated and we can obtain the grating period by measuring the diffraction angle.

The laser-induced capillary wave method has advantages in that it is possible to measure viscosity (1) over wide viscosity ranges, (2) at high speed, (3) in a contact-free manner through the use of an optical system, and (4) without the necessity of additional absorbing materials in the samples by using a pulsed CO<sub>2</sub> laser. Thus, the laser-induced capillary wave method constitutes a unique technique.

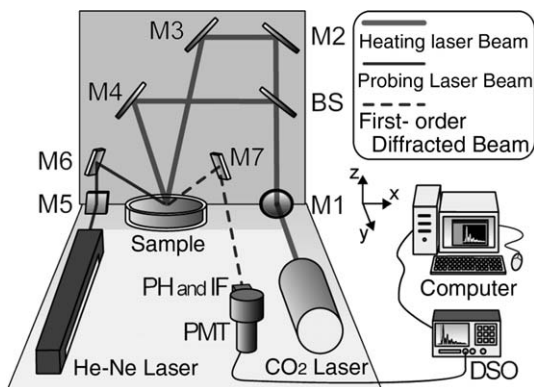


Fig. 3. Experimental apparatus for the laser-induced capillary wave method using a pulsed CO<sub>2</sub> laser.

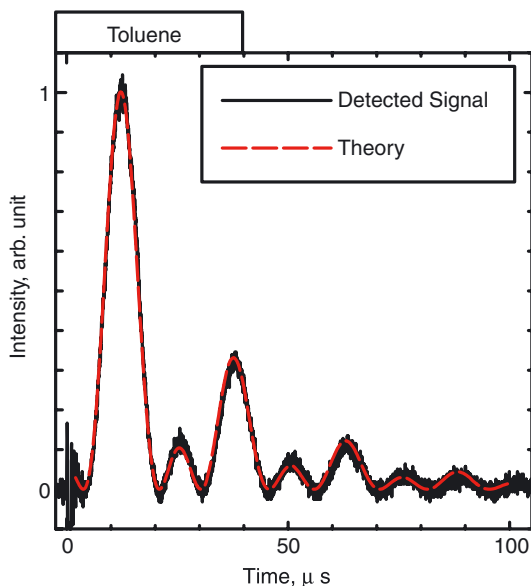
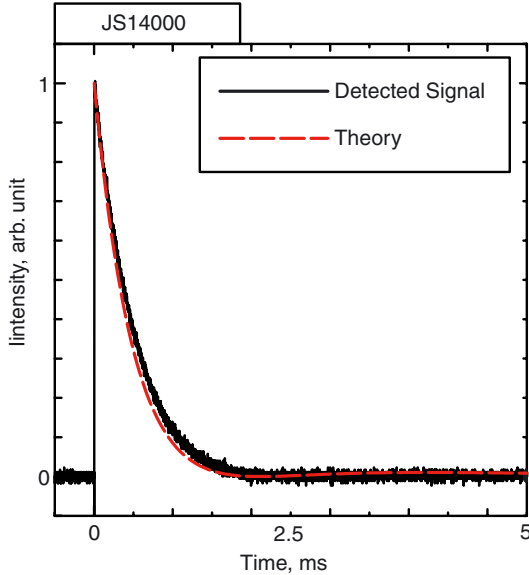


Fig. 4. Comparison of the detected signal with theoretical calculations for toluene ( $\Lambda = 50 \mu\text{m}$ ) at room temperature.

#### 4. EXPERIMENTAL RESULTS AND DISCUSSION

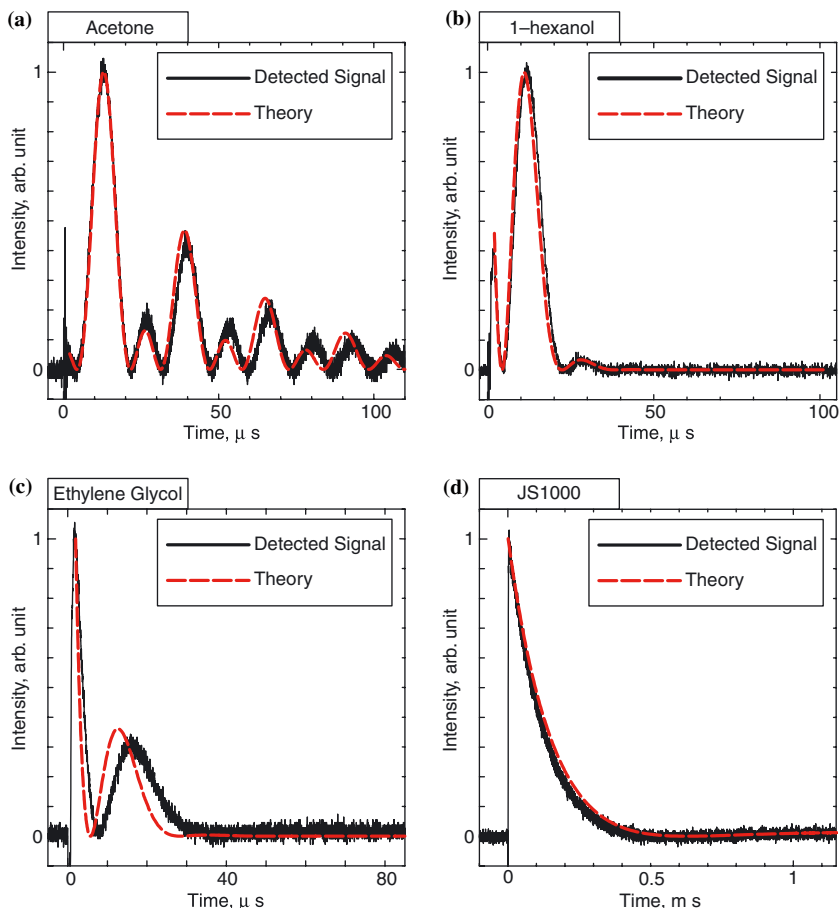
As a first step, in order to confirm the principle of the present method, we have employed pure liquids such as acetone, toluene, 1-hexanol, ethylene glycol, JS1000, and JS14000. JS1000 and JS14000 are



**Fig. 5.** Comparison of the detected signal with theoretical calculations for JS14000 ( $\Lambda = 60 \mu\text{m}$ ) at room temperature.

standard liquids for calibrating viscometers certified by the National Institute of Advanced Industrial Science and Technology (AIST) in Japan.

As an example, in Fig. 4 we show the signal detected by the present technique for toluene (whose viscosity is  $0.55 \text{ mPa}\cdot\text{s}$  at  $298 \text{ K}$ ), which is measured at room temperature (approximately  $298 \text{ K}$ ), at atmospheric pressure, and at a capillary wavelength (equal to fringe spacing of heating beams) of  $50 \mu\text{m}$ , and the theoretical waveform, which is calculated by Eqs. (5) and (11) using the thermophysical properties listed in Table I. Here, the intensity of the diffracted beam for each result is normalized at the highest peak. In the case of measuring toluene which is one of the typical low viscosity liquids, the detected signal is a damped oscillating wave. The detected signal for toluene agrees quite well with the theoretical calculation which takes into account the effect of the surface tension distribution. On the other hand, the detected signal shown in Fig. 5 is overdamped, when we measure a high viscosity sample such as JS14000 (whose viscosity is  $7080 \text{ mPa}\cdot\text{s}$  at  $298 \text{ K}$ ) under the same conditions as for toluene except at a wavelength of  $60 \mu\text{m}$ . The detected signal for JS14000, which is one of the highest viscosity liquids in the measured samples, is also in good agreement with theory which is calculated in consideration of the dependence of the thermophysical properties on the waveform. Thus, as



**Fig. 6.** Comparisons of the detected signals with theoretical calculations for (a) acetone ( $\Lambda = 50 \mu\text{m}$ ), (b) 1-hexanol ( $\Lambda = 50 \mu\text{m}$ ), (c) ethylene glycol ( $\Lambda = 60 \mu\text{m}$ ), and (d) JS1000 ( $\Lambda = 60 \mu\text{m}$ ) at room temperature.

shown in Figs. 4 and 5 we have shown the characteristic detected signals of toluene and JS14000 in comparison with theory. These remarkably different waveforms can be explained in terms of the difference of the viscosity, i.e., the viscosity of JS14000 is more than  $10^4$  times that of toluene.

Additionally, comparisons of the detected signals with theoretical calculations for acetone (whose viscosity is  $0.30 \text{ mPa}\cdot\text{s}$  at  $298 \text{ K}$ ), 1-hexanol (whose viscosity is  $4.6 \text{ mPa}\cdot\text{s}$  at  $298 \text{ K}$ ), ethylene glycol (whose viscosity is  $14 \text{ mPa}\cdot\text{s}$  at  $298 \text{ K}$ ), and JS1000 (whose viscosity is  $610 \text{ mPa}\cdot\text{s}$  at  $298 \text{ K}$ ) are shown in Fig. 6a–d in order of ascending viscosity. As

can be seen from Figs. 4 to 6, the agreement between the theory and the detected signals is excellent for six different liquids having viscosities ranging from 0.33 to 7080 MPa·s. This result is quite promising to determine the wide range of viscosity extracted from these detected characteristic waveforms.

The present study demonstrates that the laser-induced capillary wave method using a pulsed CO<sub>2</sub> laser has great potential for measurements over a wide range of viscosity, which requires neither contact with the sample nor absorbable materials in the samples. The mechanism of laser-induced capillary wave generation consists of the contributions of thermal expansion and the temperature dependence of the surface tension. A study to solve the inverse problem in order to determine the viscosity based on the detected waveforms is currently underway.

## ACKNOWLEDGMENTS

The work described in this paper has been supported in part by the Science and Technology Agency under the Promotion System for Intellectual Infrastructure of Research and Development of Japan, by a Grant-in-Aid for Japanese Ministry of Education Science and Culture (No. 09555076), and by a Grant in Aid for the 21st Century Center of Excellence for “System Design: Paradigm Shift from Intelligence to Life” from the Ministry of Education, Culture, Sport, and Technology of Japan. Professor M. Terazima of Kyoto University is gratefully acknowledged for his valuable discussions. The authors thank K. Yabui who helped with the measurements and calculations.

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