A New Project at NMIJ for an Absolute Measurement of the Viscosity by the Falling Ball Method

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A new measurement system for determining an accurate and precise absolute value of viscosity have been developed by combining nano-technologies with the classical falling ball principle. The purpose of this project at the National Metrology Institute of Japan (NMIJ) is an absolute measurement of viscosity in a high viscosity range with a relative standard uncertainty of 10^{-4} to 10^{-5} by the falling ball method, and then a determination of the absolute value on the viscosity of water with a relative standard uncertainty of 10^{-4} by the stepping-down method using the capillary viscometers, where a new absolute measurement is needed to remove the present ambiguity in the internationally accepted reference value of water. To overcome the difficulties in achieving such a reduced uncertainty, we have newly developed the systems for measuring the fall velocity by combining laser interferometric technique with image processing, and for the diameter using latest technology of the spherical interferometer, respectively. Details are given on the concept of the new method and the progress of the development with particular emphasis on the velocity measurement system.

1. Introduction

The absolute value of the viscosity of water is the metrological basis for the viscosity scale, which is used as a primary reference value for calibrating viscometers and viscosity of other fluids in the field of metrology and thermophysics. This internationally accepted viscosity values of water at 20 ℃ and atmospheric pressure as stated in ISO Technical Report 3666[1] are 1.0016 mPa·s for dynamic and 1.0034 mm^2/s for kinematic viscosity, which is based on the value reported by Swindells et al.[2] about fifty years ago. The reference value is commonly used throughout the world as the primary standard liquid for the calibration of capillary viscometers constituting the viscosity standard at the national metrology institute.

However, the evaluation of the uncertainty in the most reliable measurement by Swindells et al. was incomplete, and due to the existing discrepancies in the available data sources including other experimental data since the work of Swindells et al., the relative uncertainty of the reference value was expanded to 0.17 %. Recent result reported by Berstad et al.[3] is also reliable, but the result does not agree with the result of Swindells et al. within the uncertainty stated by the authors. And so a new absolute viscosity measurement using a recent measurement technology developed after the above reports is needed internationally.

For improving the present situation and removing the ambiguity in the reference value, NMIJ decided in 1998 to start a new absolute measurement of the viscosity of water.

In this paper, we present the concept and the target of our project, in which we have employed the classical falling ball method as the measurement principle combined with new measurement technology. The progress of the development of the new measurement system is also presented.

2. Theory of the falling ball method

When a ball falls in the Newtonian fluid of infinite extent without boundary and inertial effect, the dynamic viscosity η is related to the terminal velocity of the ball V by the following Stoke's equation

$$\eta = \frac{d^2(\rho_b - \rho_l)}{18 \cdot V} \cdot g \qquad (1)$$

where d is the diameter of the ball, ρ_b it's density, ρ_l is the density of the fluid, g is the acceleration of gravity.

In practical experiments wall and inertial effect drag the ball, causing a reduction of the fall velocity. For cylindrical tube with a diameter D, the wall effect is given by Faxen[4] as

$$V_{correct} = V(1 - 2.104\left(\frac{d}{D}\right) + 2.089\left(\frac{d}{D}\right)^3 - 0.948\left(\frac{d}{D}\right)^5 + \ldots)^{-1} \quad (2).$$

For the correction due to the inertial effect, Oseen's approximation[5] in the Navie-Stokes equation gives the following

$$V_{correct} = V(1 + \frac{3}{16}\mathrm{Re})^{-1} \quad (3)$$

where Re is the Reynolds number.

The corrections due to the higher order approximations were derived by Goldstein[6] as

$$V_{correct} = V(1 + \frac{3}{16}\mathrm{Re} - \frac{19}{1280}\mathrm{Re}^2 + \frac{71}{20480}\mathrm{Re}^3 + \cdots)^{-1} \quad (4)$$

,and Proudman and Pearson[7] as

$$V_{correct} = V(1 + \frac{3}{16}\mathrm{Re} + \frac{9}{160}\mathrm{Re}^2 \cdot \ln\frac{\mathrm{Re}}{2} + \cdots) \quad (5).$$

These correction factors to V as a function of Re are compared to each other as the differences from Stoke's approximation in Fig.1. The figure shows

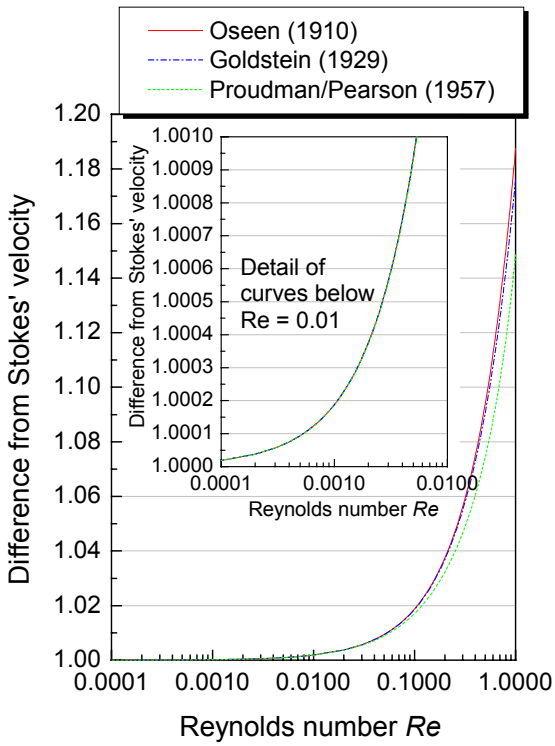

Fig.1. Comparison of the correction equations as a function of Re.

that all curves overlap within the relative deviation of less than 10^{-5} at low Re of around 0.001, which suggests that the ambiguity of the choice of the correction equations for the inertial effect may be negligible at the experimental condition of such a low Re. Considering our target uncertainty of the absolute measurement (described in the next section), the magnitude of the correction is not negligible even in this range of low Re, though the magnitude is fairly reduced.

3. Target and Concept of the Project at NMIJ

The aimed relative standard uncertainty of the absolute value of water is 10^{-4}. It can be achieved by measuring each measurement parameter with a relative uncertainty of 10^{-5}.

Before starting this project, classical methods used for measuring the liquid viscosity were all compared and evaluated[8], and we found that the classical falling ball method may be used for reducing the uncertainty of the absolute measurement by using the latest technology of the laser interferometry with nanometer measurement, particularly for the dimensional measurement of a sphere and the fall velocity measurement. On the base of that feasibility study we have set the target of this project for an absolute measurement of viscosity in a high viscosity range with a relative standard uncertainty of 10^{-4} to 10^{-5} by the falling ball method, and then a determination of the absolute value on the viscosity of water with a

Table 1. Target uncertainties in the absolute measurement of viscosity.

Uncertainty component	Value	Standard uncertainty	Relative contribution to the viscosity of water
diameter of the ball	2 mm	20 nm	2.0×10^{-5}
effective fall distance	10mm	150 nm	1.5×10^{-5}
effective fall time	10 s	100 μs	1.0×10^{-5}
temperature	20 ℃	2 mK	4.9×10^{-5}
density of the ball			2.5×10^{-5}
density of liquid sample			1.0×10^{-5}
acceleration of gravity			1.0×10^{-7}
inertial correction			1.0×10^{-4}
wall correction			1.0×10^{-4}
stepping-down method			1.0×10^{-4}
combined relative standard uncertainty of the viscosity of water			1.8×10^{-4}

relative standard uncertainty of 10^{-4} by the stepping-down method using the capillary viscometers[9]. Main difficulties in achieving the uncertainty described above are the absolute measurement of the falling velocity and the diameter of the ball. In order to overcome such difficulties, we have newly developed the velocity measurement system by combining laser interferometric technique with image processing, and the diameter measurement system using latest technology of the spherical interferometer for dimensional measurement of a silicon sphere[10], respectively.

A conceptual representation of the principle of the velocity measurement and the diameter measurement is described in Fig.2. In Fig.2 (a), the diameter of the centered sphere is measured by the phase-shifting method of the spherical interferometer, where we use single crystal silicon sphere with the diameter of 2 mm and the mass of about 7 mg as the falling ball. For the diameter measurement, the technology for the silicon sphere with the diameter of 94 mm and the mass of 1 kg had been already established at NMIJ[11]. We have considered that developing newly of the spherical interferometer lead to an uncertainty of 20 nm for the diameter measurement of the falling ball on the base of the above established technology. In Fig.2 (b), The z-scan motion of the CCD camera on the motorized stage is controlled to track the falling motion of the ball so as to keep the ball image within a few pixels in the captured frames of the camera. At the same time, a vertical displacement of the moving camera as a function of time is measured by the laser interferometer, that is synchronized to the shutter timing of the camera capturing image. In order to remove an Abbe error caused by a pitch motion of the z stage, both of the vertical and the angular (pitch) displacements of the moving camera are measured simultaneously using dual axes laser interferometer. The position of the ball can be measured with an uncertainty of about 150 nm by combing the above interferometrically measured results and the ball displacement on the CCD image obtained by the correlation analysis of the image data with a resolution higher than 1/30 pixel.

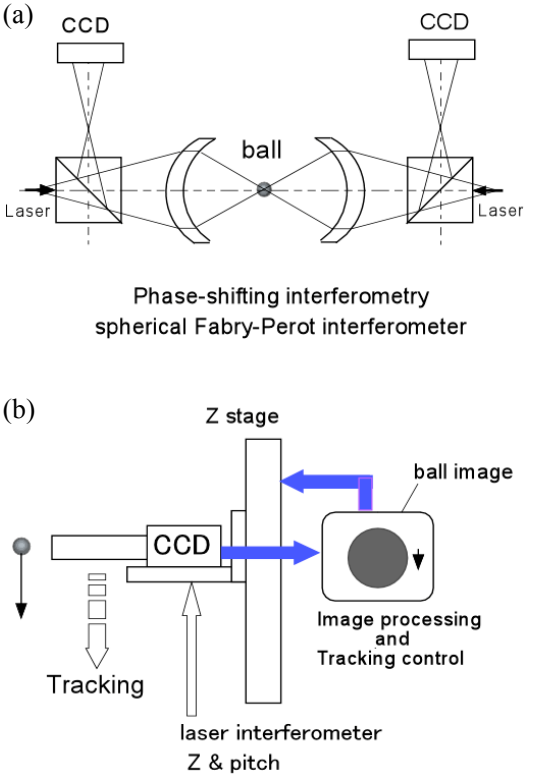

Fig.2. Principle of the viscosity measurement system under development. (a) diameter measurement system (b) velocity measurement system

The target of the uncertainty for this absolute measurement is shown in Table 1. In this project, it seems to be feasible to achieve a relative standard uncertainty of 0.02 %. Almost completing the development of elemental technologies for the falling ball method, construction of the measurement system is in progress to perform actual measurements.

4. Velocity measurement system

Figure 3 shows the schematic diagram of the whole system for the falling ball experiment under construction, which consists of a velocity measurement system and a temperature controlled bath. The velocity measurement system consists of a dual axes optical interferometer, Z stage with linear motor, CCD camera with long WD lens and a main control system using microprocessor and PC for a image processing, a time-laps data acquisition and store, and a motor control to track the ball.

A Michelson-type polarizing interferometer has been developed for measuring Z and pitch displacements of the z stage, having a hybrid polarizing beam splitter with quarter wave plates for the two-folded optical pass. Each of the light beams for z and pitch from the interferometer is divided into four beams. For interference fringe counting, phase shifted four beams (0°, 90°, 180°,

270°) by a combination of wave plates and beam splitters are detected in each of axes so that the two of the four signals with a phase difference of 180° are used as differential amplification. Resultant two output signals phase-shifted by 90° are introduced into quadrature fringe counter. A single count in the counter corresponds to 40 nm in Z displacement. For the pitch measurement, the above phase-shifted two signals are introduced to electrical interpolator for dividing each of the electrical phases into 1/25, which results the total resolution of 0.03 " in the measurement of the angular displacement.

Since a motion of the ball is simple and the reproducibility of the motion is extremely high, particularly when falling the ball in temperature controlled bath (< 0.1 mK), it is considered that the vision based tracking does not necessarily need a real-time image. The precision of the tracking can be improved by refining a programmed motion so as to reduce the ball displacement on the image within a few pixels by the analysis of recorded images through the repeated fall.

To combine the interferometrically obtained data and correlation results of the image analysis, a displacement magnitude on the image must be calibrated to the actual vertical and angular displacement of the camera on the z stage, respectively. For that purpose, the calibration apparatus have also been developed using a precision tilt stage.

The temperature controlled bath has a structure of vacuum insulation surrounded by a circulated temperature controlled liquid. A sample cell, having an automatic control mechanism for release-retrieval of the falling ball, is immersed into the inner liquid inside the bath, temperature of which is controlled by PC through monitoring temperature using a standard platinum resistance thermometer and the thermometry bridge.

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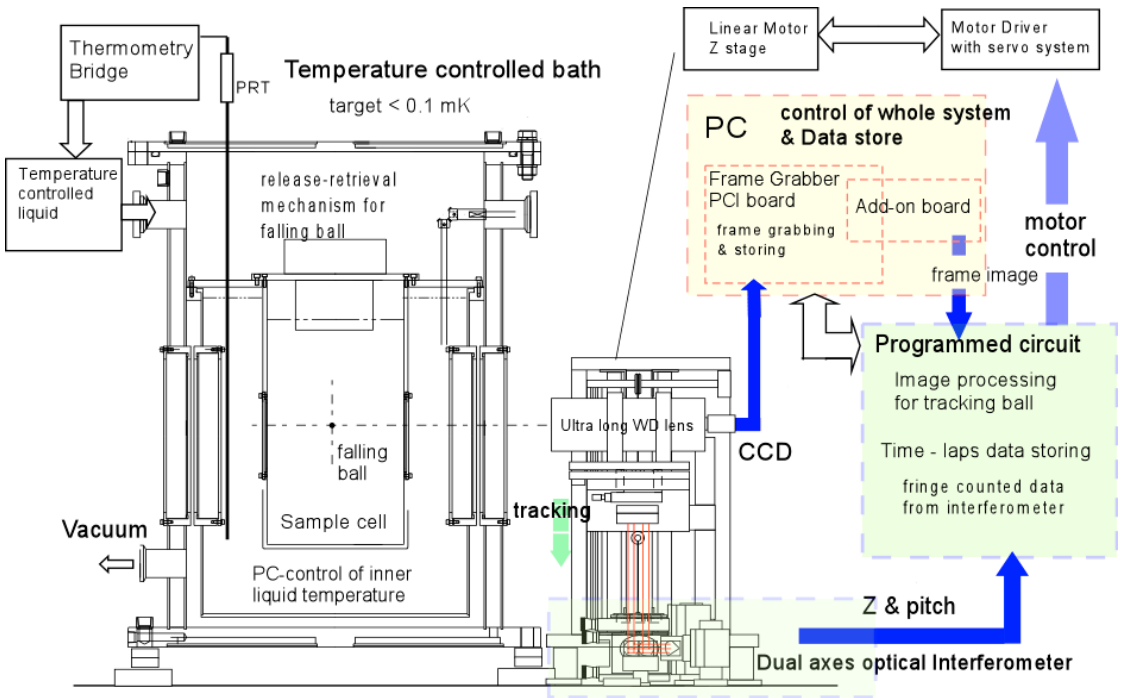

Fig.3. Schematic diagram of the fall velocity measurement system under construction.