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An oscillating cup viscometer based on Shvidkovskiy algorithm for molten metals

Ping Zhu[a,1], Jiahui Lai[a,1], Jing Shen[b], Kesen Wu[a], Lili Zhang[b,*], Jian Liu[a,*]

[a] School of Control Science and Engineering, Shandong University, Jinan 250061, China
[b] School of Mechanical and Automotive Engineering, Qilu University of Technology, Jinan 250353, China

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ABSTRACT

In this paper, an oscillating cup viscometer based on Shvidkovskiy algorithm and time-resolved reflection measurement was described for the molten metals over a broad temperature range up to 1800 K. The viscosity of molten metals was automatically calculated from Shvidkovskiy formulae by obtaining the time period and the logarithmic decrement of the oscillations. The representative viscosity measurements using our homemade viscometer were performed for water at a room temperature and pure aluminum in a temperature range from its melting point to 1300 K. It was found that the obtained viscosity results showed good agreement with the previous reports for both water and aluminum, indicating its reliable viscosity measurement. In addition, the experimental uncertainty of the viscometer was also estimated by analyzing the errors propagation of the input parameters of Shvidkovskiy equation. The results showed that a very low uncertainty of 2.1% for the dynamic viscosity measurement, which attributed from the small errors of the logarithmic decrement and time period with highly accurate time-resolved reflection measurement. Therefore, the developed oscillating cup viscometer would be an effective tool for high temperature viscosity measurement in both basic research and industrial applications.

1. Introduction

The viscosity of molten metals plays an important role for both scientific research and industrial applications [1]. However, it is a difficult task to measure the viscosity of liquid metals due to their high melting points, low viscosities and chemical activity [2]. Although a number of methods can be considered to measure the viscosity of molten metals, oscillating cup technique has been proven to be the most appropriate [1,3,4]. This kind of viscometer focuses on the viscosity of Newtonian liquids and has been successfully used to determine the viscosities of numerous liquid metals, including pure metals (e.g., Al [5], Sn [6]) and metal alloys (e.g., Al-Cu [7], Fe-Co [8], Al-Cu-Si [9]).

For an oscillating cup viscometer, specimen is placed in a crucible suspended by a torsion wire to form a pendulum. Then the pendulum is set in torsional oscillation about a vertical axis. This motion is gradually damped due to the frictional energy absorption and dissipation within the viscous liquid. The liquid viscosity can be determined by observing the decrement and the time period of the oscillations [4,10,11]. The merits of the oscillating cup technique are its mechanical simplicity, ease in creating inert atmosphere and inactive materials, small specimen volumes allowing stable temperature profiles, and absolute method etc. [12]. More importantly, this technique has the ability to accurately measure the time period and the amplitude decay, which enables precise viscosity determination [2,5,10].

Since the 1960s, many of research groups worldwide have showed interest in this type of viscometer. At the same time, a variety of approximation algorithms have proposed to measure the viscosity of liquids at high temperature. Knappworst [13], Shvidkovskiy [14], and Roscoe [15,16] provided a class of solution models and working equations for oscillating cup viscometers. The working formulae included the correction factors and had the advantages of formulae simplicity and easy to understand [5]. These techniques, especially Roscoe analysis, are still commonly employed nowadays [1,17,18]. Subsequently, a complete analytical model and relevant working equation (eliminating the correction factor) were described by Kestin, Newell and Beckwith [19,20]. It was claimed that this model was more accurate and comprehensive than that of Roscoe, and thus became the subject of further investigation [2,21,22]. A deal of chemical engineers have adopted this method for deriving the viscosity of molten materials [5,10,23,24]. However, due to the elimination of the correction factor,

* Corresponding authors.
E-mail addresses: sdilizll@163.com (L. Zhang), lj@sdu.edu.cn (J. Liu).
[1] These authors contributed equally to this work.

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Backwith-Newell model was almost helpless to the deviations originating from the instrument itself or the measuring process [5]. From this, in the face of achieving reliable viscosity measurement, the input parameters of the completely analytical algorithm were usually required to measure with rather high precision.

Despite numerous successful working formulae and oscillating cup viscometers have been reported, there are still several challenges to be overcome. One of the major difficulties is that various apparatus and viscosity estimation methods often yield different experimental results even for melts of pure elements [1,2,10]. For instance, the viscosity of pure aluminum (Al) at melting point reported by Wang et al. [5] was 1.38 mPa s, while it was given as 1.1 mPa s at another literature survey [25]. Obviously, there are still some discrepancies between laboratories for the viscosity of Al at melting point. Therefore, the performance of oscillating cup viscometer needs to get further improved, in the aim to obtain reliable viscosity data of molten metals.

The work described in this paper aims to produce an oscillating cup viscometer using Shvidkovskiy equation with the correction factor. A way to improve the performance of the viscometer was enhancing the accuracy of measurement time. The viscosity measurements were carried out on water at room temperature and Al in the temperature range between 1300 K down to the solidification. Moreover, the experimental uncertainty of the developed instrument was also estimated.

2. Description of the viscometer apparatus

The instrument developed in this study is a torsional oscillating cup viscometer for high temperature molten metals. This viscometer is composed of an oscillating system, a heating system, a vacuum system, and a system for laser oscillation detection (Fig. 1). In the oscillating system, a stepping motor at the top of the device is attached to the upper end of a torsion wire to initiate the oscillations. The lower end of the wire is fixed to a mirror holder and an inertia disc which is connected to a rigid rod. The rigid rod is designed to have a small moment of inertia and offer a good heat barrier. Screwed to the lower end of the rigid rod is an alumina crucible of 29 mm inner diameter and 60 mm height. The specimen is placed in the non-conducting crucible after carefully treatment. For the heating system, the main component is a high frequency induction electric furnace which is used to heat the specimen inside the crucible during measuring process. The

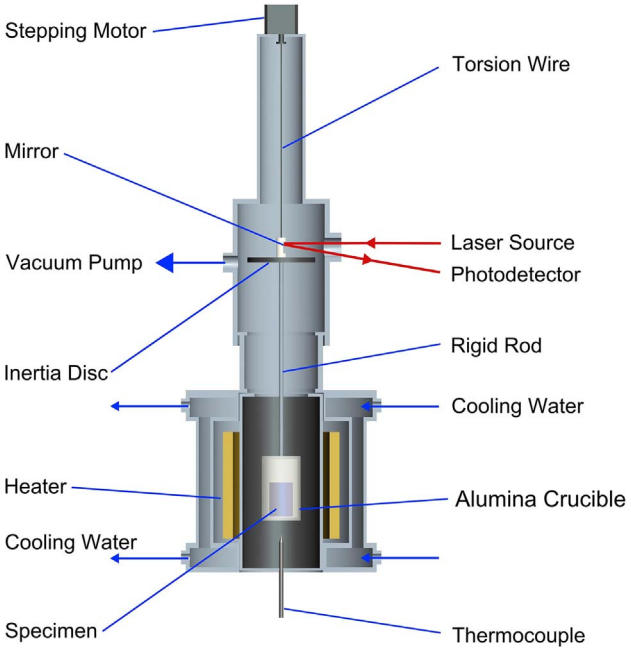

Fig. 1. Schematic view of the oscillating cup viscometer.

temperature of the furnace is measured with a thermocouple which is held in the bottom and middle exterior of the crucible. The precision of the temperature measurement is better than ± 2 K. During experimental process, making use of a vacuum pump, the space of the furnace is cleaned to vacuum. Then it is filled with argon at constant pressure to prevent the samples from being oxidized at high temperature.

Laser oscillation detection is accomplished by the measuring system, which is shown schematically in Fig. 2(A). The measuring system is composed of a He-Ne laser, two photodetectors, a homemade data acquisition and control system, a computer to control and analyze the data, and a temperature control unit. The laser source is located about 2 m from the mirror mounted on the suspension system. The distance between the photodetector A and B is 20 cm. Firstly the stepping motor is driven through the computer to initial the oscillations of the cup. Following that a laser light is directed to the mirror, and the reflected light from the mirror is focused as a small spot on the photodetectors (Fig. 2(B)). Then the phototransistors in the detector complete the transformation of the light to electric signals. Thereafter, the electric signal is processed to generate the time interval data by the data acquisition and control system. It is demonstrated that the time detection accuracy of this system can reach up to 0.1 ms, which helps to improve measurement precision of the time period and logarithmic decrement of oscillations. Next, the successive time interval signal is directly transmitted to the PC through an RS232 serial interface. The last step is to analyze the collected time interval data to obtain the period and logarithmic decrement of the oscillations and the final viscosity result.

All operations of the viscometer apparatus, including experimental parameters setting, motor control, data storage and analysis, and display of temperature–viscosity curve, are controlled via a homemade software.

3. Data processing and analysis

For the measurement, a cylindrical cup containing a fluid is set in a free but damped oscillation. The motion of the cup is given by Eq. (1) [1,21]:

$$I\left(\frac{d^2\Phi}{dt^2}\right) + L\left(\frac{d\Phi}{dt}\right) = -D\Phi \tag{1}$$

Therein, I is the moment of inertia of the whole pendulum, t is the absolute time, and D is the force constant of the torsion wire; Φ refers to any small angle displacement of the fluid from the equilibrium; the damping parameter L denotes a function of the viscosity and density of the fluid, the inner radius of the cup and the height of the liquid sample in the cup.

Based on the second-order differential equation, many of suitable formulae have been proposed for viscosity calculation. Among them, methods requiring correction factors are more prevalent. Therefore, here we will stick to the Shvidkovskiy equation [26] in the following. The kinetic viscosity ν is given by Eq. (2):

$$\nu = \frac{1}{\pi}\frac{I^2(\delta - T\delta_0/T_0)^2}{(mr)^2 T\sigma^2} \tag{2}$$

where

$$\sigma = 1 - \frac{3}{2}\gamma - \frac{3}{8}\gamma^2 - a + (b - c\gamma)\frac{2nr}{h} \tag{3}$$

T is the time period of the oscillations and T_o is that of the empty cup, δ and δ_0 represent the logarithmic damping decrement of the cup with and without specimen respectively, m is the mass of the liquid sample, r is the inner radius of the cup and h is the height of the liquid sample in the cup, $\gamma = \delta/2\pi$, n is the number of solid planes contacted horizontally by the liquid sample (i.e. in the case of a vessel having the lower end closed and its upper surface free, n = 1, if the vessel encloses the fluid in top and bottom, n = 2; in this study n = 2), and a, b, c are