Silikáty *27*, s. 333—341 (1983)

Laboratorní a výpočetní technika

HIGH-TEMPERATURE OSCILLATION VISCOMETER

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Received 29. 10. 1982

The high-temperature oscillation viscometer constructed by the authors operates at up to 1 700 °C in the viscosity range from 0.1 to 1.5 Pa . s. The viscosity is calculated from the value of the logarithmic decrement obtained by measuring the time intervals of a moving beam using two fixed photodetectors. The output of the measuring device was connected 'on-line' to a programmable minicomputer which processed the measuring data. The bodies used in the viscosity measurements were either cylinders or cylinder with conical ends. The viscometer was tested on aqueous glycerine solutions using equations for movement of a cylindrical body in a viscous medium, whereas for the cylindrical body with conical ends the equation was corrected by calibration in glycerine solutions. The absolute error of the viscosity determinations was less than 3 %. Viscosity measurements were carried out on melts having the compositions $CaSiO_3$, $CaMgSi_2O_6$ *and* $Ca_2MgSi_2O_7$ *in the temperature interval 1 390 to 1 630 °C.*

INTRODUCTION

The oscillation viscometer was used in the determination of viscosity of molten salts in a number of studies [1]—[8]. It has found application in particular at low-viscosity liquids, volatile liquids and in the measuring of viscosity of gases [7], [9], [10]. The selection of this type of viscometer for viscosity measurements on oxide melts was given by the relatively low viscosities of the melts in question (melts with the CaO/SiO_2 modulus of about 1, steelmaking slags, etc.) as well as by the high consumption of platinum metals involved in the use of a rotary viscometer.

The use of a cylindrical measuring body, which enables to obtain absolute viscosity values [2], was restricted because of the possibility of a bubble forming at the bottom surface of the measuring body. This is why the alternate shape of a cylinder with conical ends was employed. Movement of such a body through a viscous medium has not so far been described by an equation which would allow viscosity to be calculated directly. For this reason calibration by means of aqueous glycerine solutions had to be employed with bodies of this shape.

The oscillation viscometer is based on the principle of measuring the damping of torsion oscillations of a symmetrical body of defined shape, suspended on a thin torsion wire and immersed in the liquid being measured. Viscosity is calculated from the logarithmic decrement defined as the difference of the logarithms of two subsequent amplitudes, when knowing the density of the liquid being measured, the body dimensions and the moment of inertia of the entire suspension system performing the damped harmonic oscillations.

APPARATUS

The design of the oscillation viscometer for work in air atmosphere up to 1 700 °C is shown in the schematic diagram in Fig. 1. The measuring bodies were alternatively a cylinder whose diameter was equal to its height (5.47 ± 0.01 mm) and a cylinder with conical ends with equal cylinder radius, cylinder height and heights of both cones (3.718 ± 0.005 mm). The measuring bodies and the parts of the suspension system situated in the furnace hot zone were of the PtRh40 alloy. The parts of the suspension system outside the hot furnace zone were of stainless steel or brass. For determining and change in the inertia of the suspension system three rings of brass and one of stailess steel weer used, which had been machined with the most high precision.

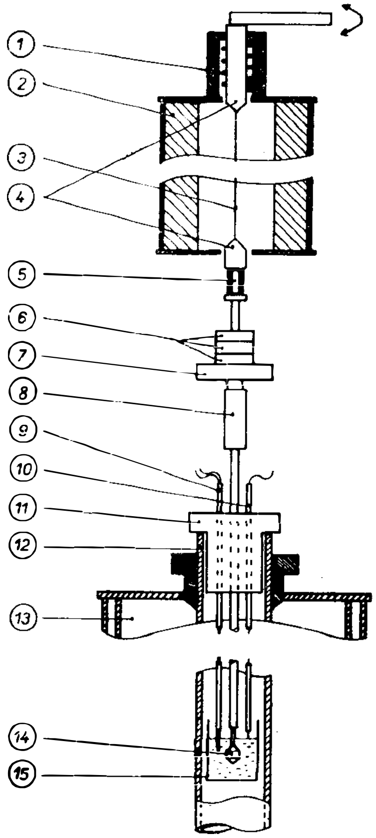

Fig. 1. Schematic sectional view of the oscillation viscometer. 1 — winding device, 2 — tempered protective jacket of the torsion wire, 3 — torsion wire, 4 — gripping collets, 5 — mirror, 6 — additional brass rings, 7 — heavy ring of stainless steel, 8 — bayonet letting down device, 9 — thermocouple, 10 — level surface contact, 11 — furnace closure, 12 — corundum furnace shaft, 13 — water-cooled furnace, 14 — measuring body, 15 — crucible with melt.

All the parts of the suspension system were machined to a tolerance of ±0.01 mm and after assembly the system was aligned so that the maximum deviation would not exceed 0.1 mm. Special care was paid to the machining of the measuring bodies whose surface was finally polished. The length of the suspension system from the torsion wire to the measuring body was about 85 cm and its weight was 497.5 g.

The torision fibre was a dia. 0.3 mm PtW8 wire about 60 cm in length. The PtW8 alloy is recommended in [11] for its low internal friction and a high stability of its torsional properties. The fibre was suspended in a cylinder tempered at 30 °C, to the top of which was fitted a mechanical winding divice serving for shifting the suspension system from its equilibrial position. The fibre ends were clamped in a way ensuring perfect alignment of the suspension system. To relieve internal stresses, the wire was annealed for 2 hours at about 1 200 °C by passing controlled AC while loading the wire with 500 g.

The moment of inertia of the suspension system was determined by measuring the change in the period due to addition of brass rings, the moment of intertia of which was calculated by the equation

$$I = \Delta I \left(\frac{t^2}{t^2 - t_0^2} \right) \tag{1}$$

where ΔI is the moment of inertia of the brass ring added, t and t_0 are the periods of the system with and without the brass rings respectively. Table I lists the measured values of the moment of inertia with the individual rings added, and also the moment of inertia of the whole suspension system.

Table I

Moment of inertia of the suspension system with the measuring body in the form of a cylinder with conical ends. The period without the additional rings, $t_0 = 4.6146$ s

Ring No.	$\Delta I_i \times 10^5$ / kg m²	t_i / s	$I_i \times 10^5$ / kg m²	$I_0 \times 10^5$* / kg m²
1	0.5315	4.8342	5.985	5.454
2	0.5309	4.8333	6.002	5.471
3	0.5312	4.8341	5.985	5.454
1 + 2	1.0624	5.0429	6.532	5.469
1 + 2 + 3	1.5936	5.2434	7.068	5.474

The mean value $I_0 = (5.464 \pm 0.009) \times 10^{-5}$ kg m²
*) $I_0 = I_i - \Delta I_i$

The furnace employed in the high-temperature measurements was provided with double molybdenum winding (dia. 0.9 mm wire) on a corundum tube of 50 mm ID and 60 mm OD and 550 mm in length. The furnace winding was protected from oxidation by a forming gas. The furnace was fed from a NOCONTA AC tyristor power source using the TRS 97 proportional controller so that the relative temperature stability was within ±1 °C. The temperature was measured with a PtRh6/PtRh30 thermocouple whose hot joint was placed in the liquid being measured. The thermocouple had been calibrated at the melting points of gold, nickel and palladium. Over the temperature range of 1 200 to 1 700 °C the temperature was measured with an error of ±5 °C. The melt was placed in a crucible of PtRh30 alloy, 40 mm in diameter and 50 mm in height. The amount of the substance weighed into the crucible was such that the melt volume amounted to about 40 cm^3.

The damped harmonic oscillations of the suspension system were recorded by means of a halogen lamp beam reflected from the suspension system mirror and received by two KP 101 phototransistors situated in its trajectory. The detectors were placed roughly symmetrically to the oscillation axis and their mutual distance was 5 cm. The distance between the lamp and the detectors and the suspension system mirror was about 1 m, the angular displacement at the maximum amplitude amounted to about 6 deg. The time intervals between the phototransistor pulses were measured with an accuracy to 10 μs. The overall accuracy of the oscillation measurement was ±1 ms with the given arrangement. The set of time intervals was transmitted trough an interface into the memory of the EMG 666 minicomputer. The mathematical processing of the set of time intervals, the computation of the period the amplitudes and the logarthmic decrement was described in [4].

Equations for the computation of the viscosity of liquids measured by the oscillating sfere and cylinder (with equal diameter and height) were derived in [2, 12]. For the cylinder, the equation has the form

$$\eta = \frac{2\delta I}{5\pi R^3 t_0}\left(\frac{1}{2 + b_1 R + P}\right), \tag{2}$$

where

$$P = \frac{b_1 R + 1}{(b_1 R + 1)^2 + b_1^2 R^2}; \qquad b_1 = \sqrt{\frac{\varrho\pi}{\eta t}};$$

η is viscosity (Pa . s), ϱ is density of the liquid being measured (kg m^{-3}), R is the radius of the measuring cylinder (m), I is the moment of inertia of the torsion pendulum (kg m^2), t is the period in the liquid being measured (s), t_0 is the period in vacuo (s) and δ is the logarithmic decrement. For a sphere, equation (2) has an analogous form [12] where the value of the constant is 3/4 and not 2/5. It is to be assumed that the size of this constant depends on the geometric shape of the measuring body and that with one in the shape of a cylinder with conical ends, it should be possible to use equation (2) with the value of the constant determined by calibration measurements.

The logarithmic decrement δ, arising in equation (2), is a magnitude characterizing the damping of the torsion oscillations due solely to friction between the measuring body and the liquid being tested. The experimentally established value δ_{exp} has therefore to be corrected for the damping due to friction of the suspension system with the ambient atmosphere (δ_{air}) and for the damping due to friction of the immersed part of the rod connecting the suspension system with the measuring body (δ_{conn}). The latter correction is sigificant namely when using measuring bodies of small sizes. The corrected logarithmic decrement value is then calculated from the equation

$$\delta_{\text{corr}} = \delta_{\text{exp}} - \delta_{\text{air}} - \delta_{\text{conn}}. \tag{3}$$

The logarithmic decrement values were calculated by processing 10 to 20 periods in dependence of the viscosity of the liquid being measured, while the first 3 to 6 periods from starting the damped oscillation were not recorded. This time was found satisfactory for equalization of the whole suspension system after its starting.

EXPERIMENTAL

The calibration solutions were prepared from A.R. glycerine (Lachema, Brno) in concentrations of 100, 98, 94, 92, 90 and 85 wt. %. The density values of the solutions required for viscosity calculations were taken from ref. [13]. The solutions were tempered in a thermostat.

Specimens having the compositions $CaSiO_3$, $CaMgSi_2O_6$ and $Ca_2MgSi_2O_7$ were prepared by calcination of $CaCO_3$, A.R., and $MgCO_3$, A.R., Lachema, Brno and subsequent fusion with SiO_2, A.R. Erba, Milano in the corresponding stoichiometric ratio. The density values of the melts were taken over from ref. [14].

In the viscosity measurements proper, use was made of the mean moment of inertia value of the suspension system, 5.464×10^{-5} kg m^2. The moment of inertia of the suspension system including the measuring body of cylindrical shape was 5.420×10^{-5} kg m^{-2}. For viscosity measurements at high temperatures the effect of temperature on the moment of inertia of the suspension system was determined; this effect is caused by thermal expansion of the measuring body. It was found that the change in the moment of inertia of the system with temperature was obviously smaller than the error with which the moment of inertia was determined. Calculation based on the thermal expansion data indicated that the change in the moment of inertia for 1 500 °C amounted to the order of 10^{-9} kg m^2.

The temperature dependence of the dimensions of the measuring body was determined on the basis of thermal expansion measurements on the PtRh40 alloy employed. The following equation was used for correcting the measuring body radius with respect to thermal expansion in the temperature interval of 1 200 to 1 700 °C:

$$R = (3.7055 \times 10^{-3} + 5.0185 \times 10^{-8}\vartheta), \qquad [\mathrm{m}] \qquad (4)$$

where ϑ is temperature in °C.

The logarithmic decrement value in air at room temperature was $1.05 \pm 0.1 \times 10^{-3}$. The period in air amounted to about 4.62 s in the given arrangement.

Table II lists the measured and literature values of viscosity of aqueous glycerine solutions. The values are means of 5 to 6 measurements. The measurements were carried out at 20 °C.

The viscosity of the $CaSiO_3$, $CaMgSi_2O_6$ and $Ca_2MgSi_2O_7$ melts is shown in Fig. 2. The viscosity values are means of 5 to 6 measurements. The experimental

Table II

Measured and literary viscosity values of aqueous glycerine solutions at 20 °C

Glycerine wt. %	η/Pa . s				$\Delta\eta$* / Pa . s
	ref. [16]	ref. [17]	ref. [18]	exp.	
100	1.412	1.499	—	1.453	0.009
98	0.939	0.974	0.997	0.947	0.005
94	0.437	0.458	0.458	0.436	0.006
92	0.310	0.328	0.326	0.315	0.002
90	0.219	0.235	0.233	0.226	0.004
85	0.109	0.113	0.112	0.113	0.003

*) Standard deviation of experimental measurements.

values were treated by the least squares method. Viscosity of the melts in question can be described by the following exponential equations:

$$\eta_{CaSiO_3} = 8.981 \times 10^{-6} \exp(18931/T); \qquad \Delta\eta = 2.1\,\%, \tag{5}$$

$$\eta_{CaMgSi_2O_6} = 7.230 \times 10^{-6} \exp(19840/T); \qquad \Delta\eta = 1.6\,\%, \tag{6}$$

$$\eta_{Ca_2MgSi_2O_7} = 1.441 \times 10^{-5} \exp(16820/T); \qquad \Delta\eta = 2.5\,\%. \tag{7}$$

DISCUSSION

In the designing of the oscillation viscometer, use was made of experience gained in the construction and operation of the apparatus decribed in [2], [4] to [7], [15]. The design was influenced by the less exacting precision requirements in this instance. Application of the oscillation viscometer for temperatures of up to 1 700 °C and for oxide melts with viscosities of the order of 0.1—1 Pa s requires scaling down of the measuring body dimensions and increasing the moment of inertia of the suspension system, which necessarily leads to a lower sensitivity of the instrument and consequently to a lower measuring accuracy. In view of this the new viscometer shows parameters roughly 5-times less favourable than the presently top-grade oscillation viscometer [5] which, however, has so far been used at temperatures up to about 1 100 °C, exhibiting an absolute measuring error of 0.5—1 % for routine measurements at this temperature [6].

The results of viscosity measurements on aqueous glycerine solutions involve an estimated absolute measuring error lower than 3 %. Table II shows a comparison of the data measured with literary data at 20 °C and indicates that the latter exhibit differences of up to 9 %. With regard to an analysis of the methods employed, the data given in [16] were considered the most reliable ones. Those specified in [17, 18] are higher by about 3 to 5 %. It may be said, however, that

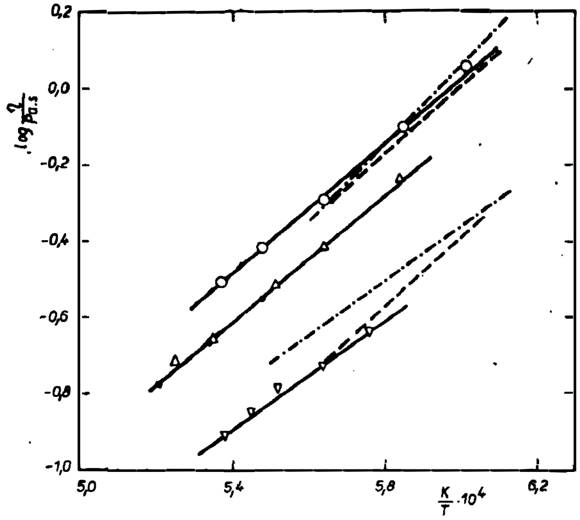

Fig. 2. Viscosity of the melts of $CaSiO_3$, $CaMgSi_2O_6$ *and* $Ca_2MgSi_2O_7$. ○ — $CaMgSi_2O_6$, △ — $CaSiO_3$, ▽ — $Ca_2MgSi_2O_7$, -.-.-. *after [19]*, ——— *after [20]*, ● — *after [21]*, *full lines according to equations (5)—(7).*

the viscosity values of the respective solutions are in agreement with the literary data [16] within the 3 % absolute measuring error, even though the standard deviation throughout the whole series of measurements in the present study was lower than 3 %.

The results of viscosity measurements on $CaSiO_3$, $CaMgSi_2O_6$ and $Ca_2MgSi_2O_7$ melts were compared with the values measured in studies [19]—[21]. Fig. 2 shows the temperature dependence of viscosity according to [19]—[21] obtained by interpolation of the concentration relationships. The measurements in [19] used a rotary viscometer with a graphite crucible in a nitrogen atmosphere. For the $CaMgSi_2O_6$ melt, a good agreement was obtained with the values given in [19], while for the melt of $Ca_2MgSi_2O_7$ the values given in [19] are higher by about 21 %. The measurements performed in [20] used an oscillation viscometer with a cylindrical measuring body and the melt placed in a platinum crucible. The viscosity falues for $CaMgSi_2O_6$ given in [20] are lower by about 7 %. For $Ca_2MgSi_2O_7$, the viscosity values are higher by about 3 % than the results of the present study. Viscosity of the $CaSiO_3$ melt in [21] was measured with a rotary viscometer, and its results are identical with those of the present study within the framework of the experimental error.

An analysis of errors involved in viscosity measurements with an oscillation viscometer is discussed in detail in [4], [5], [15]. The recommended measurement of viscosity on water was impossible in the present case because the maximum sensitivity had been adjusted for the viscosity range from 0.1 to 1.5 Pa . s.

The greatest source of errors of the present viscometer was the determination of the measuring body diameter and the error in the determination of the logarithmic decrement. The measuring body diameter was measured with a cathetometer with an accuracy of ±0.005 mm. This uncertainty is responsible for the viscosity error of 0,78 %. An estimate of the error due to uncertainty in the logarithmic decrement determination was calculated from its standard deviation, the maximum value of which was 5×10^{-5}. This value includes also measuring of logarithmic decrement in air, when the swing of the torsion pendulum takes place. The mentioned error in the logarithmic decrement determination is responsible for the viscosity uncertainty of up to 0.8 % depending on the actual viscosity value. The lesser errors are due to inaccuracy in the determination of the moment of inertia of the suspension system, in the measuring of the period and in the determination of density of the liquid being measured. The standard deviation involved in the determination of the moment of inertia of the suspension system, 9×10^{-8} kg m^2, causes an error of 0.17 % in the viscosity value. The oscillation period was measured with an accuracy of ±1 ms, which is responsible for an error of 0.06 % in the viscosity value. The same error, 0.06 % of the viscosity value, is caused by density determination described in [14], which is accurate within ±0.005 g . cm^3. The sum of the errors mentioned above gives the value of the estimated total error of 1.87 % for viscosity measurements on the viscometer described. Further minor errors can obviously occur as a result of imperfect thermal stabilization of the torsion fibre, non-uniform immersion of the measuring body, etc. The standard deviation of the temperature dependence of viscosity of the melts in question was lower than 3 % in all the instances, which is in a good agreement with the above estimate of the measuring error.

The authors dedicate this paper to the memory of Ing. I. Votava.

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VYSOKOTEPLOTNÝ OSCILAČNÝ VISKOZIMETER

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Skonštruoval sa vysokoteplotný oscilačný viskozimeter pracujúci do teploty 1700 °C v oblasti viskozít 0,1—1,5 Pa . s. Schematický prierez viskozimetra je uvedený na obrázku 1. Viskozita sa vypočíta z hodnoty logaritmického dekrementu, získanej meraním časových úsekov pohybujúceho sa lúča medzi dvomi fixovanými fotodetektormi. Výstup meracieho zariadenia bol pripojený „on-line" na programovateľný kalkulátor, ktorý spracovával výsledky merania. Pre meranie viskozity sa použilo alternatívne teliesko v tvare valca, alebo valca s kužeľovitými koncami.

Funkcia viskozimetra sa overila meraním viskozity vodných roztokov glycerínu pri použití rovníc pre pohyb valcového telesa vo viskóznom prostredí, zatiaľ čo pre valcové teliesko s kužeľovitými koncami sa táto rovnica korigovala pomocou kalibrácie v roztokoch glycerínu. Stanovenie viskozity sa uskutočnilo s absolútnou chybou merania menšou ako 3 %.

Stanovila sa viskozita tavenín zloženia $CaSiO_3$, $CaMgSi_2O_6$ a $Ca_2MgSi_2O_7$ v teplotnom rozmedzí 1390—1630 °C. Teplotná závislosť viskozity meraných tavenín sa popísala exponenciálnymi rovnicami:

$$\eta_{CaSiO_3} = 8{,}981 \,.\, 10^{-6} \,.\, \exp\,(18\,931/T);\ \Delta\eta = 2{,}1\,\%$$
$$\eta_{CaMgSi_2O_6} = 7{,}230 \,.\, 10^{-6} \,.\, \exp\,(19\,840/T);\ \Delta\eta = 1{,}6\,\%$$
$$\eta_{Ca_2MgSi_2O_7} = 1{,}441 \,.\, 10^{-5} \,.\, \exp\,(16\,820/T);\ \Delta\eta = 2{,}5\,\%$$

ВЫСОКОТЕМПЕРАТУРНЫЙ ОСЦИЛЛАЦИОННЫЙ ВИСКОЗИМЕТР

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Был собран высокотемпературный осциллационный вискозиметр, работающий до температуры 1 700 °С в области вязкостей 0,1—1,5 Па . с. Схематическое сечение вискозиметром приводится на рис. 1. Вязкость рассчитывали на основании величины

логарифмического декремента, полученной измерением промежутков времени движущегося луча между двумя неподвижными фотодетекторами. Выход измеряющей установки подключили „on line“ к програмируемому калкулятору, обрабатывающему результаты измерения. Для измерения вязкости использовали альтернирующее тело в виде цилиндра или цилиндра с конусообразными концами.

Работу вискозиметра проверяли измерением вязкости водных растворов глицерина, применяя уравнения для движения цилиндрического тела во вязкой среде, в то время как для цилиндрического тела с конусообразными концами это уравнение корректировали с помощью калибрации в растворах глицерина. Определение вязкости проводили с абсолютной погрешностью измерения, недостигающей 3 %.

Устанавливали вязкость расплавов состава $CaSiO_3$, $CaMgSi_2O_6$ и $Ca_2MgSi_2O_7$ в температурном интервале 1 390—1 630 °C. Температурную зависимость вязкости измеряемых расплавов описывали с помощью экспоненциальных уравнений:

$$\eta_{CaSiO_3} = 8{,}981 \,.\, 10^{-6} \,.\, \exp\,(18\,931/T);\ \Delta\eta = 2{,}1\,\%$$

$$\eta_{CaMgSi_2O_6} = 7{,}230 \,.\, 10^{-6} \,.\, \exp\,(19\,840/T);\ \Delta\eta = 1{,}6\,\%$$

$$\eta_{Ca_2MgSi_2O_7} = 1{,}441 \,.\, 10^{-5} \,.\, \exp\,(16\,820/T);\ \Delta\eta = 2{,}5\,\%$$

Рис. 1. Схематическое сечение вискозиметром: 1 — натяжная установка, 2 — темперированное защитное покрытие крутильного волокна, 3 — крутильное волокно 4 — клещи, 5 — зеркало, 6 — добавочные латунные кольца, 7 — тяжелое кольцо из нержавеющей стали, 8 — штыковое пусковое устройство, 9 — термопара, 10 — контакт, служащий для определения уровня, 11 — затвор печи, 12 — корундовая шахта печи, 13 — водоохлаждаемая печь, 14 — измеряющее тело, 15 — тигель с расплавом.

Рис. 2. Вязкость расплавов $CaSiO_3$, $CaMgSi_2O_6$ *и* $Ca_2MgSi_2O_7$. O — $CaMgSi_2O_6$, △ — — $CaSiO_3$, ▽ — $Ca_2MgSi_2O_7$, — . — . — . — *согласно* [*19*], ----- *согласно* [*20*]. ● — *согласно* [*21*], *сплошные линии согласно уравнениям* (*5*)—(*7*).