Software for oscillating-cup viscometry: verification of data reasonableness and parametric identification of rheological model

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Abstract. The general model of the oscillating-cup viscometry is presented. The methods of the parametric identification of flows of non-Newtonian fluids are developed and programly implemented. The theory is used to study the rheological behavior of liquid metals.

The oscillating-cup viscometry (e.g. [1–3]) is prevalent technique for measurement of the properties of metal melts. In practical applications, the using of the theoretical works is difficult foremost due to complicated numerical adaptation of them. Only for calculations in the frames of the exact solutions known from the middle of the last century, the simplified relationships are frequently used. An analysis of complex problems, (for example, the extending of traditional assumptions [2]), described by nonlinear nonsteady partial differential equations is not usually carried out in a practice. In this connection, the software for estimation of the viscometer motion characteristics, the schemes and models to correct the data interpretation at the step «oscillation parameters – fluid properties» have been developed.

The mathematical model of an experiment includes following equations that we represent in terms of physical components.

1) The scalar mass conservation equation:

\[
\frac{\partial \varrho_r}{\partial t} + \frac{\varrho_r}{r} \frac{\partial \varrho_z}{\partial z} = 0.
\]  

2) The vector momentum conservation equation:

\[
\rho \left( \frac{\partial \varrho_r}{\partial t} + \frac{\partial \varrho_r}{\partial r} + \frac{\partial \varrho_r}{\partial z} \right) = -\frac{\partial \varrho_p}{\partial r} + \frac{\partial \sigma_{rr}}{\partial r} + \frac{\partial \sigma_{rz}}{\partial z} + \frac{\sigma_{rr} - \sigma_{\varphi \varphi}}{r};
\]

\[
\rho \left( \frac{\partial \varrho_\varphi}{\partial t} + \frac{\partial \varrho_\varphi}{\partial r} + \frac{\partial \varrho_\varphi}{\partial z} \right) = \frac{\partial \sigma_{\varphi r}}{\partial r} + \frac{\partial \sigma_{\varphi z}}{\partial z} + \frac{2 \varrho_\varphi}{r};
\]
\[ \rho \left( \frac{\partial \vartheta_z}{\partial t} + \vartheta_r \frac{\partial \vartheta_z}{\partial r} + \vartheta_z \frac{\partial \vartheta_z}{\partial z} \right) = -\frac{\partial \varrho}{\partial z} - \rho g + \frac{\partial \sigma_{rz}}{\partial r} + \frac{\partial \sigma_{zz}}{\partial z} + \frac{\sigma_{rz}}{r}, \]  
(4)

where \( \vartheta_r, \vartheta_\varphi, \vartheta_z \) are the radial, azimuthal and axial components of the velocity vector \( \mathbf{V} \); \( g \) is the free fall acceleration; \( \rho \) is the pressure; \( r, z \) are the radial and axial coordinates \((r = 0 \text{ and } z = 0 \text{ on the cylinder axis and on the bottom, respectively})\); \( t \) is the time; \( \sigma_{ij} \) is \( i j \)th component of the extra stress tensor \( \sigma \); \( \rho \) is the fluid density; in equations (1)–(4), the flow is axially symmetric, \( \rho = \text{const} \) are taken into account.

3) The tensor rheological constitutive equation.

As an example of rheostable fluids, the Bingham model is considered:

\[ \sigma = (2\nu' + \sigma_0 / II_{\mathbf{D}}) \mathbf{D} \text{ for } II_{\sigma} \geq \sigma_0; \]
\[ \sigma = \mathbf{D} \text{ for } II_{\sigma} < \sigma_0, \]
(5)

and, as an example unsteady-state fluid, the upper-convected Maxwell model:

\[ \mathbf{\sigma} + \lambda \frac{\partial \mathbf{\sigma}}{\partial t} = 2\nu \mathbf{D}. \]
(6)

The model of Newtonian fluids is

\[ \mathbf{\sigma} = 2\nu \mathbf{D}. \]
(7)

In equations (5)–(7), \( \mathbf{D} \) is the tensor of deformation rates; \( II_{\mathbf{A}} = (\sum A_{ij}A_{ij}/2)^{1/2} \) is the second invariant of a certain tensor \( \mathbf{A} \); \( A_{ij} \) is \( i j \)th component of \( \mathbf{A} \); \( \lambda \) is the relaxation time; \( \nu \) is the kinematic coefficient of viscosity; \( \nu' \) is the viscosity after yielding; \( \sigma_0 \) is the yield stress.

4) The oscillation equation for the cylinder:

\[ \ddot{\varphi} = \frac{P_c}{K}, \]
(8)

where \( \varphi \) is the angular displacement of the cylinder from equilibrium; \( K \) is the moment of inertia of the empty cup system with respect to cylinder axis; \( P_c = P_0 + P_c + P_\tau \) is the total torque of all external forces acting on viscometer about rotation axis; \( P \) is the torque applied to the cylinder from a fluid:

\[ P = -2\pi R^3 \int_0^H \sigma_{\varphi \varphi} dz + 2\pi R \int_0^H \left[ \left( \sigma_{z\varphi} - (a-1)\sigma_{z\varphi} \right)_{\partial z} \right] \right] dA; \]
(9)

\( a \) is the number of viscometer end-walls: \( a = 1 \) for a fluid with a free surface at \( z = H \); \( a = 2 \) for full cylinder with a lid; \( P_0 = P_c + P_\tau \) is the torque acting also on the empty cup system; \( P_c \) is the elastic torque of the suspension torsion; \( P_\tau \) is the resistance torque (due to the resistance in the surrounding air and the internal friction of the suspension wire); for a small oscillation, it is possible to write:

\[ P_0 = 2\delta_0 \dot{\varphi}/\tau_0 + N\dot{\varphi}/K; \]
(10)

\( P_\tau \) is the varying external action, for example \( P_\tau = P_0 \sin \omega t \); \( P_a, \omega \) are an amplitude and an angular frequency of driving torque; for damped oscillations \( P_\tau = 0 \); \( N \) is the stiffness coefficient of the suspended wire; \( H, R \) are the height and radius of a cylindrical specimen; \( \delta_0, \tau_0 \) are the logarithmic decrement and the period without the fluid; while an overdot denotes a time derivative.

5) The initial conditions (for damped oscillation regime):

\[ t = 0: \ \vartheta_r = \vartheta_z = \vartheta_\varphi = 0; \ a) \ \alpha = \alpha_0, \ \frac{d\alpha}{dt} = 0, \ \alpha_0 \sim 6^\circ, \ b) \ \alpha = 0, \ \frac{d\alpha}{dt} \neq 0 \ (\sim \alpha_0), \ c) \ t \in (0, t_0): \ P_\tau \neq 0 \ \text{for a) or b)}, \ t \geq t_0: \ P_\tau = 0. \]
(11)

The boundary conditions:

\[ r = 0: \ \vartheta_r = \vartheta_\varphi = d\vartheta_z / \partial r = 0 \ (\text{by symmetry}); \ r = R: \vartheta_r = \vartheta_z = 0, \ \vartheta_\varphi = \dot{\varphi}R \ (\text{no-slip condition}); \]
\[ z = 0: \ \vartheta_r = \vartheta_z = 0, \ \vartheta_\varphi = \dot{\varphi}r; \ z = H: \ \vartheta_r = \vartheta_z = 0, \ \vartheta_\varphi = \dot{\varphi}r \ (a = 2) \]

and

\[ \frac{\partial \vartheta_\varphi}{\partial z} = \vartheta_\varphi = \frac{\partial \vartheta_\varphi}{\partial z} = \vartheta_\varphi = 0 \ (a = 1). \]
(12)
in the presence of a wall slip, a difference between \( \partial \varphi \) and \( \alpha r \) depends on the stress.

This program module enables to model a fluid motion in the viscometer and to study an evolution of oscillations under different initial conditions, a spatial flows at \( \varphi = 0 \) (for example for a larger values \( \alpha_0 \)) including an appearance of instability. Besides the axially symmetric model \((1)-(12)\) (version 1) the soft includes the computations also for two basic particular cases: for traditional account of the vector \( V \) components: \( \varphi = 0 \) (e.g. \( [2, 3] \)) (version 2), and for the infinitely long cylinder: \( \partial F / \partial z = 0 \), where \( F \) is the component of \( V \) or \( \sigma \) (version 3). Then, for instance, in version 1 for the zero initial conditions, the nonzero \( \sigma \) components are \( \sigma_{y0}, \sigma_{z0}, \sigma_{\varphi0} \) for which equation \((6)\) takes the form: \( \sigma_{ij} + \lambda \sigma_{ij} = 2\nu D_{ij}, \sigma_{\varphi0} + \lambda (\sigma_{\varphi0} - 4(\sigma_{y0} D_{y0} + \sigma_{z0} D_{z0})) = 0 \). Here \( \sigma_{\varphi0} \) can be used in equation \((2)\) for determination of the pressure field, and the equation \((3)\) is of traditional sense. Numerical solution is carried out by finite-volume method with SIMPLER-algorithm in version 1, the Barakat-Clark method in version 2, the method of straight lines in version 3. Following dimensionless variables are used (see also \([3–5]\)):

\[
A = \pi \frac{HR^3}{2K}, \quad \xi_0 = \frac{R}{d}, \quad \chi_0 = \frac{H}{R}, \quad \text{Bm} = \sigma_0 = (\nu \rho H D) \quad \text{We} = \frac{2 \pi q_0}{\tau_0}, \quad d = (v q_0)^{1/2}, \quad \theta = q_0 / q, \quad (13)
\]

where \( A, \xi_0, \chi_0 \) describe the basic experiment conditions; \( \xi_0, \chi_0, \text{Bm}, \text{We} \) describe the viscous, plastic and elastic fluid properties; \( \text{Bm, We} \) are the Weissenberg and Bingham numbers; \( q \) is the oscillation frequency of the filled viscometer.

The main features of the motion of a viscometer for non-Newtonian fluids were noted in \([4–6]\). In the general case, the oscillations are non-isynchronous and their parameters (the logarithmic decrement \( \delta \) and the period \( \theta \)) depend on the half-period number \( n \) (for a linear fluid, only in the transient) (figure 1). In the transient, except for the base harmonic, a non-periodic components are presented in the law of oscillations, and the average value of \( \delta \) increases for linear (curve 1 in figure 1) purely viscous fluids. Also, a periodic components exist in the presence of elasticity. The behavior of nonlinear rheostable fluids in viscometer is interpreted in terms of the effective viscosity \( \nu_{ef} \) and of the values \( \xi_{0ef} \) (for example, for equation \((5)\)): \( \xi_{0ef} = \xi_0 (\nu + \sigma_0 (2 \rho H D)^{-1})^{-1/2} \). For a fluid with elasticity, the parameter \( \text{We}_{ef} \) is introduced also. The dependence of the oscillation parameters on \( \xi_0, \text{We} \) and an experiment conditions was noted for linear fluids in \([2, 4]\): for viscous fluids, the period \( \theta \) decreases when \( \xi_0 \) increases, \( \delta = \delta(\xi_0) \) has a maximum at \( \xi_0 = 4.3 \) (if \( \chi_0 \to \infty \) and above this value (for smaller \( \chi_0 \))), and a number of extremums in \( \delta = \delta(\xi_0) \) and \( \theta = \theta(\xi_0) \) increases while as the elastic properties become stronger. Thus, \( \delta = \delta(n) \) passes maximum for a visco(pseudo)plastic fluid (curve 2) if \( \xi_{0ef} > \xi_{0ef} \) (\( \theta \) increases) and for a dilatant fluid if \( \xi_{0ef} < \xi_{0ef} \) (\( \theta \) decreases). There is part with \( \delta = \delta_0 \) at small \( \alpha \) if \( \text{Bm} \neq 0 \). A nonlinear fluids with an elasticity demonstrate an oscillating character of \( \delta = \delta(n) \) and \( \theta = \theta(n) \) (curve 3).

![Figure 1](image1.png)  
**Figure 1.** The dependence \( \delta = \delta(n) \).

![Figure 2](image2.png)  
**Figure 2.** The distribution of the rigid zones in flow.
The viscous penetration depth is proportional to $v_{ef}$, it is $\sim 10d$ for equation (7), and for a rheostable fluids, this depth changes accordingly. In the presence of elasticity, the velocity profile is less convex, the developed flow region is larger. The rigid zones $I$ (figure 2a) arise in a viscoplastic fluid near the cylinder wall at each $n$ moves toward the core 2, and their radius increases as $n$ increases. If the distribution of the stress along the cylinder height is nonuniform (the model (1)–(5), (8)–(12)), then in the rigid zone, the cavities near flowing zone which are closed at $a = 2$ can appear (figure 2b).

For practical applications, the numerical computations are not effective, and another block has to be applied. Here, the solutions are based on the exact solutions for the linear viscous and viscoelastic fluids. The block includes following: 1) for Newtonian fluid, the program modules for a data interpretation by a simplified and exact relationships [2, 3] and simultaneous estimation of the viscosity and density of Newtonian fluids; 2) the software for the prime and inverse viscometry problems for Newtonian and non-Newtonian fluids. The packages for a system identity testing, construction and minimization of a performance criterion, selection of an optimal experiment conditions and formation of a representation of the input data, calculation of a statistical characteristic of a parameters, etc., are attached.

In the prime task, the parameters identification is carried out from the performance criterion formed by $\xi_{0ef}$ for every $n$ when the known expressions for $H_D$ for the linear fluids [2, 3] at $\xi_{0ef}$ are used. In the inverse task, the transient oscillation stage is taken into account by the relationships for Newtonian fluids (for the linear viscoelastic fluids, in terms of a complex viscosity (e.g. [4])) [3]. Under practical experiment conditions, the computation results obtained by this method and by the numerical models for the axially symmetric flow and with traditional account of the velocity components, coincide within the scope of the fluid property observability and within the limits of measurement accuracy of the oscillation parameters and numerical computations (more correct than $10^{-2}$) out of the transient. Using the schemes developed, the features of a motion of a viscometer filled by a fluid of any rheological model can be easily revealed: in particular, the features of a rheostable fluids can be described in terms of Newtonian model of the axially symmetric flow.

The software was used for the data reasonableness verification of by means of the simultaneous measurement of $\nu$ and $\rho$ [7] and for the identification of a rheological behavior of liquid metals in the experiments with the oscillating-cup viscometer. For instance, the features of the non-Newtonian character are observed under small values of the investigated temperature range including the heterogeneous zone between the solidus and liquidus (subsided as the temperature $T$ increases) and around the anomalies on the temperature curves $T = T(\nu)$. For example, after melting in the experiments with iron, the number $B_m \sim 0.3$ and the viscosity after yielding is of the same order as the Newtonian viscosity for larger $T$ ($\nu \sim 0.005$ poise). Interpreting the data within the scope of the Ostwald–de Waele model, the flow behavior index was found to be $\sim 0.65$. In the estimation of the Newtonian model, the viscosity $\nu \sim 0.2$ poise corresponds to the high-viscosity region [2] ($\xi_0 \sim 2$).

Software is implemented in FORTRAN. Some programs are available on http://physics.susu.ru.

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