The high-pressure viscometer up to 400MPa with primary method of viscous moment measurements

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Abstract
A new high-pressure high-shear stress viscometer for pressure up to 400MPa, Couette type (rotational), has been developed. The wide shear rates range and shear stress can be applied for investigations of non-Newtonian liquids. The moment of viscous forces, acting in measuring viscosity cylinders, are measured outside of high-pressure chamber using primary method or using moment (force) sensors. That was realized by use of two counter rotating drive shaft systems with computer controlled stepped motors. As a pressure source a 10:1 intensifier with high-pressure manganin sensor were used.

1. Introduction
Lately many experimental works concerning rheological properties of high viscosity liquids (lubricants and biologic substances) under high pressure has been published [1, 2]. The main problems, that have to be resolved here are the realization of the well defined measurement flow (knowledge about shear rates and shear stresses distribution) and measurements of viscous forces (their moments) in order to find fundamental relation between shear tress and shear rate in all points of measurement flow. In the case of Couette’s type of flow – rotational type viscometer – those problems concentrate on defining the distribution function of particle velocities in the clearance and measurement of moment acting on chosen measurement cylinder. For this last task, the resistance strain gauge [3] or optic techniques [4] were used. In this paper a novel solution for viscous moment measurement is proposed [5].

2. Construction of viscometer
In figure 1 a sketch of the main details of viscometer, with extended active measurement surface, is shown. The principle of this device states two, nominal identical in geometry and materials, counter rotating systems, that compensate friction moments in moving seals and bearing systems (located outside of the pressure chamber). One can conclude that on pressure vessel acts moment being a result of viscous friction of investigated liquids only. Relatively small friction forces moment of the pressure chamber bearing system (see “3” in figure 2) is easy recorded.

Being independent of pressure and measured viscosity it can be eliminated as systematic error. Essentially higher force friction moments are connected with moving seals. Two counter winded identical elastic capillaries pressurize the pressure vessel, also counter winded so, some observed rotate angle of pressure vessel is a function of viscosity friction moment only. That can be used as measuring moment parameter or can be compensated fixing pressure vessel in the constant initial position by outside moment, being in principle primary method of viscous moment measurement. Other solutions realized by authors are the use of force sensors, placed on known distance from the center of pressure chamber.

In this solution, very flexible capillary connection of pressure chamber with the pressurized system have to be use. Rotating movement is realized using two steps motors regulated by computer system with a program specially developed for presented viscometer. Initially, by a certain time resulting from difference in diameters between the measuring cylinder end diameter of drive rod, see figure 1), the measuring cylinder does not move, friction moments in sealing and bearings are compensated, so position of pressure vessel should be not changed or indications of force sensors should be not
changed. After that definite time interval, the measure cylinder will begin to rotate and observed changes of pressure vessel position (or change of force sensors) is a measure of inner viscous moment. Typical measurements characteristics are shown in right upper corner in figure 2.

Figure 1. Cross-section of main details of double rotated viscometer (first version). $F_1$, $F_2$ – internal forces, $M_1$, $M_2$ - external moments, $M_3 = M_1 - M_2$ - viscous moment measured directly outside of the pressure device.

Figure 2. Cross-section of the second version viscometer (simplified measurement system). Explanation of elements see text. On the left upper a photo of prototype and on the right upper typical measurements characteristics are shown.
In figure 2 a simplified cross-section of viscometer (and photo) is shown. As we can see, the whole viscometer can be rotated around horizontal axis, perpendicular to pressure chamber axis, by ±90°. That allowed us to use other methods of viscosity measurement as Stokes’ or falling body method. In figure 2 numbers have a following meanings: 1- stepped motors, 2- compensating (left) drive shaft element, 3 – very low friction bearing system, 4 – closure nut of pressure chamber, 5 – closure plug with moving seals (O-ring and anti extrusion teflon rings), and static seals (O-ring and Bridgman II uncompensated surface metallic ring), 6 – measuring cylinders, 7- pressure inlet, 8 – driving rod, 9 – pressure chamber, 10 – active (right) drive shaft element, 11 – thrust bearing system, 12 – viscometer chassis, allowing it rotation along horizontal axis by ± 90°.

3. Measuring system

For the system shown in figure 1 the nominal diameters of measuring, moving, cylinder are 2R₁ = 18 and 2R₂ = 20mm. The length (L) of it may range from 10 to 50mm. The nominal clearances (H) were planned to be as high as (10 – 15) μm. Parameters S defined as a ratio of clearance diameters (outside and inner one) - D₀ / D₁, is equal to 1,001, it means that velocity particle gradient value in clearances can be defined as ratio of relative cylinders velocity (V) and clearance (H) as in simple Newton flow and to be constant with errors no higher than 0.5% [6]. Finally we have used following expression: for shear stress - τ = M/2ΠH(L₁² + L₂²) and for clearance strain rate dγ/dt = 2ΠRN/H where M is viscous moment, R = 0.25(D₀ + D₁), L = length of measurement moving cylinder, N - number of rotates per second. Strain rates are calculated for both clearances separately and for viscosity calculation a mean value is taken. If stresses does not strongly depend on strain rate such approximation is acceptable. On other hand one clearance system have to be used, see figure 2.

Nominal clearance values are of two- three orders higher than dimensions of investigate liquid particles (natural vegetables oil rod type molecules have length of some nm) and one order higher than dimensions of it hypothetic conglomerates which have been detected during light scattering investigations [7].

A rheological measurement is by its nature not isothermal. In paper [8], an extensive review of the literature and analysis of viscous heating in Couette shear is presented. The ends and temperature effects (the last one, in the case of Couette type viscometers, is really very small) were taking under consideration in preliminary degree only. We assumed that its influence essentially does not change the observed phenomenon i.e. dependence of viscosity upon pressure and specially its increase by of three orders of magnitude during the pressure induced phase change [9]. In order to check the correctness of viscometer, measurements of standard viscosity liquids have been carried out. Its nominal standard values were in good agreement with obtained measurements results. The corrections for values of clearances due to pressure deformation, having influence on measurements results, had to be taken under consideration.

Data of viscometer: pressure range - (0 - 100)MPa, shear rate - (0 - 10⁻³)s⁻¹, shear stress limit 5MPa, viscosity measurement range - 1Pas - 500kPas, temperature range - (0 – 100)°C; a 6 cm³ sample is required.

4. Problem of castor oil viscosity measurement

Recently, a change of viscosity of castor oil during its high pressure phase change was investigated using capillary method [9]. The phenomenon of viscosity of high pressure castor oil phase appeared to be typical for no-time dependence thixotropic non-Newton liquid. Its apparent viscosity is strongly dependent on deformation rate as we can see in figure 3. The viscosity before transformation at pressure about 350MPa was of order of 100Pas according to literature data whereas after phase change the viscosity for deformation rate of 0.1s⁻¹ is of three orders higher. At deformation rate of order 10s⁻¹ it decreases to value of 5kPas. The values μ₀ and μ₇ for castor oil high-pressure phase were not determined yet.

Using castor oil as a pressure transmitting medium and also as a good lubricant at longer time, we should take into consideration the effects connected with its phase change, not only the giant increase of viscosity but also changes of volume, dielectric and optic properties.
Figure 3. The dependencies of apparent viscosity of castor oil high-pressure phase vs. shear rate parameter (upper line) [9] and for high-pressure rapeseed oil phase.

Paper [10] presents data of viscosity castor oil measurements using ultrasonic method of measurements using Bleustein-Gulyaev waves. It is difficult to discuss obtained there results because high-pressure castor oil phase is, most probably, a tixotrophic liquid, contains long, rod typed, molecules with structural viscosity for which, values of shear rates, play essential role. Moreover, the measurement results of viscosity of normal state castor oil do not fulfill Barus’ formula except at pressures higher than 300MPa. Comparison of “capillary viscosity”, “ultrasonic viscosity” and preliminary results obtained using described here device is presented in figure 4.

Figure 4. Schematic presentation of viscosity of castor oil during pressure induced phase changes. Continuous lines according to [9] and “ultrasonic viscosity”, dashed lines, according to [10]. ⭐ – preliminary data obtained using described here viscometer.
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