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OPTOVISCOSIMETRY OF A LIQUID FILM BY THE METHOD OF A DISPERSED MAGNETIC SUBLAYER

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A magnetooptical method of measurement of viscosity, which is based on recording the optical response (induced by the magnetic-field pulses) of a two-layer system resulting from the sedimentation of ferroparticles dispersed in the liquid under study, has been proposed. The procedure has been described and the results of measurement of the rheokinetics of a number of media, including polymer compositions formed into a thin film, in heating, evaporation, and polymerization have been presented.

Investigation of the rheological characteristics of media whose viscosity can change by many orders of magnitude in the processes of polymerization or drying under the action of high pressure, radiation, or temperature is of manifold theoretical and practical interest [1, 2]. Under such conditions, one can by far not always or with great difficulty perform measurements using the standard rheometric equipment. Such a problem is faced, in particular, in production of dispersion-distributed composite materials from a liquid-dispersed magnetic composition hardened in the presence of an external magnetic field [3].

With the aim of determining the regime of action of the field, ensuring the formation of a prescribed volume-ordered disperse structure in the material produced, we carry out computer modeling of the behavior of a system of interacting particles in a polymer matrix with a viscosity rising in the process of hardening.

To study the kinetics of viscosity of a liquid in the process of its heating, polymerization, and evaporation we have employed the magnetooptical method. Theoretical prerequisites for its use for rheological measurements have been analyzed in [4], where consideration has been given to the processes of formation of structures and kinetics of optical response under the action of a magnetic field in a thin layer of liquid covering a sublayer of spherical soft-magnetic particles (the sublayer results from the sedimentation of the latter). It has been shown that the viscosity of the liquid can be determined in this case from the rate of rise of the system's light transmission as a result of excitation of the particle sublayer by the pulses of the magnetic field initiating the process of formation of chain structures and transverse relative to the layer's plane.

The rate of change of the coefficient of light transmission of the suspension along the direction of the lines of force of the magnetic field β is in proportion to the square of the field intensity H and in inverse proportion to the liquid viscosity η:

$$\beta = kH^2/\eta \, , \tag{1}$$

where k is the coefficient determined by the sensitivity of the photoelectric measuring system. Thus, when the amplitude of the pulse of the magnetic field acting on the specimen is known, to measure the viscosity we are to determine in the experiment just the rate of change of the light transmission of the layer.

The method has been implemented on a setup whose diagram is presented in Fig. 1. The liquid under study 1 with soft-magnetic particles (nickel particles of size (10 ± 1) μm) introduced into it is placed in a cell formed by thin glass plates 2. A cylindrical spacer 3, whose thickness determines the thickness of the liquid layer formed (usually 100–300 μm), is installed along the perimeter of the plates. The cell is placed in the working gap of the inductor of a uniform magnetic field. A pulse generator 5 and a current amplifier 6 are installed in the feed circuit of coils 4 (in

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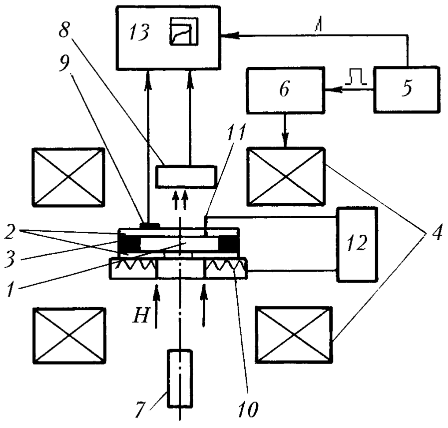


Fig. 1. Diagram of the setup.

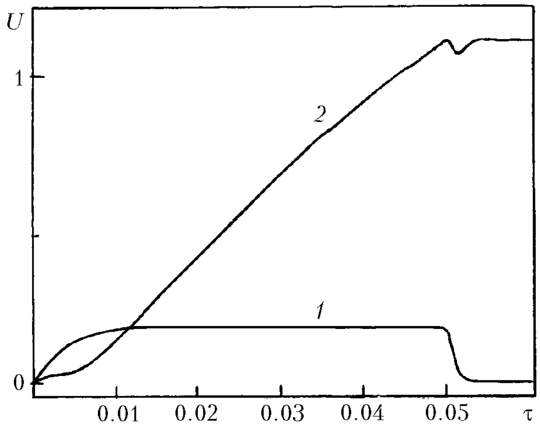


Fig. 2. Example of the change in the magnetic field (1) and the optical response of the system (2). U, V; τ, sec.

this setup, we employ Helmholtz coils). The photoelectric system of recording of the change in the light transmission of the layer consists of laser 7 and photodetector 8. The intensity of the magnetic field acting on the specimen is recorded by the Hall probe 9. The liquid under study is thermostatted by a thermal-regulation system incorporating heater 10 built into a copper disk with a hole at the center, thermocouple 11, and temperature regulator 12. The system ensures an accuracy of $\pm 0.1^{\circ}$C of the prescribed temperature (0–140°C) maintained in the liquid under study. The signal from the photodetector and the Hall probe is fed to the input of computer 13 connected to the port.

The order of the measurement is as follows. The necessary temperature of the layer of the investigated liquid is prescribed and maintained using the thermal-regulation system. At the necessary instant of time, a pulse of prescribed duration is fed to the current amplifier from the generator of square-wave signals. Simultaneously, the sync pulse from the generator triggers an analog-to-digital converter. The current pulse formed by the amplifier arrives at the inductor coils, and a pulsed magnetic field acts on the specimen. The duration of the front of the magnetic-field pulse is no longer than 10^{-2} sec for a magnetic-field intensity to 300 Oe. Structural changes in the particle layer and corresponding changes in light transmission are recorded by the photodetector. Time-base sweeps of change of the signals from the photodetector and the Hall probe are recorded on the screen and in the storage of the computer.

Figure 2 gives the characteristic oscillograms of change of the magnetic field and the optical response of the system (MS-20 oil, H = 220 Oe). The curve mapping the change in the light transmission of the layer with time is linear after the field pulse reaches its maximum amplitude, which corresponds to the results of numerical modeling of the kinetics of light transmission of the layer, caused by the aggregation of particles under the action of a magnetic-field pulse [4].

Evaluation Test of the Method. The first series of experiments has been conducted with the aim of checking the legitimacy of the use of relation (1) for determination of the liquid viscosity under the action of magnetic-field pulses heterogeneous in duration and intensity and for determination of the calibration constant k. In this series, the object of investigation was MS-20 oil into which we introduced carbonyl-nickel particles of dispersity (10 ± 1) μm. The thickness of the liquid layer in the measuring cell was set equal to 200 μm. In all the experiments described below, the mass of the introduced particles ensured a degree of filling of the layer s = 1.25. A temperature of 20°C was maintained in the measuring cell. The amplitudes of magnetic-field pulses of duration 10–50 msec varied in the range 30–130 Oe. In the indicated range of variation of the parameters of the field pulses, the calibration constant retains its constant value with a variance no higher than 3–4%. The results of the experiments on the influence of the intensity of magnetic-field pulses are presented in Fig. 3. The average value of the calibration constant (k = 2.14) is independent of the amplitude of the field pulses. The value of the dynamic viscosity of oil, which has been determined in all the experiments conducted with the above conditions with the use of relation (1), differs from the tabulated value [5] by no more than 4–5%.

To assure ourselves that it is possible to study the temperature dependences of viscosity we carried out measurements in the same index liquid (MS-20 oil) in the range of variation of temperatures 20–100°C. The results of the

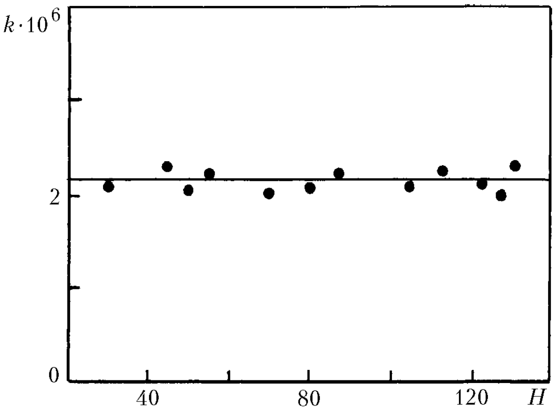


Fig. 3. On determination of the calibration constant. H, Oe; k, P·V/(Oe^2·sec).

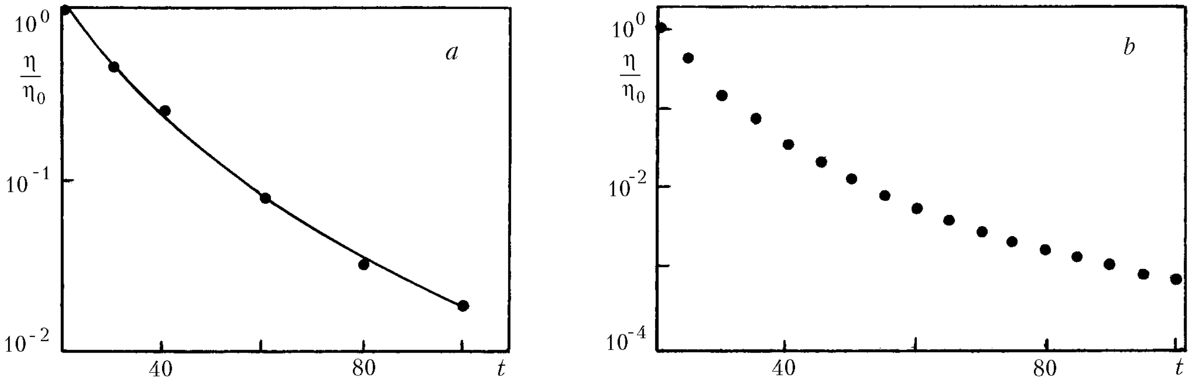
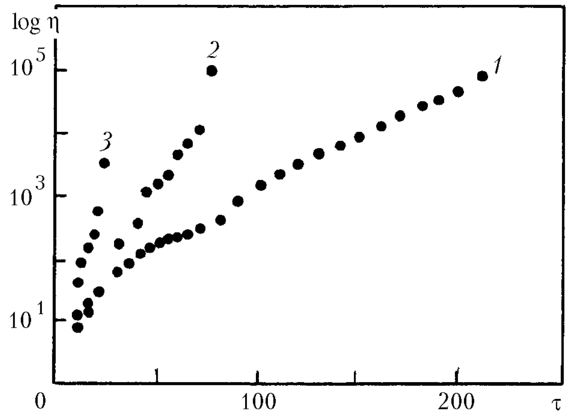


Fig. 4. Temperature dependence of the viscosity of MS-20 oil (a) (points, tabulated data [5]) and epoxy resin (b). t, °C.

experiments are presented in Fig. 4a. Here η_0 is the value of viscosity at 20°C and η is the viscosity of the oil at the temperature t. A comparison to the tabulated data of [5] shows that the values of the viscosity of oil, obtained throughout the experimental range of variation of temperature, differ little from the tabulated values and the maximum measurement error amounts to no more than 7%.

Measurement of the Temperature Dependence of the Viscosity of Epoxy Resin. Figure 4b gives the results of measurement of the viscosity of epoxy resin as a function of the temperature. As a result of magnetooptical probing, we have found that the viscosity of the medium changes 2525 times in heating from 20 to 100°C. Cyclic heating and cooling of the liquid under study do not change the behavior of the temperature dependence of the viscosity of epoxy resin.

Investigation of Rheokinetics in the Process of Hardening of Epoxy Resin. Epoxy materials are thermally reactive. The rate of the chemical reaction of crosslinking of oligomer molecules on introduction of a hardener is low at low and room temperatures and exponentially rises with further increase in the temperature. The method in question is efficient in measuring the kinetics of viscosity in the process of rapid hardening of oligomer compositions at considerable temperatures. Under the action of a pulse (with a duration to 50 msec) of the field of selected intensity (H = 220 Oe), the displacement of particles from their initial position does not exceed 5 μm even for a minimum initial viscosity of the system under study. As the viscosity increases, we can increase the value of the field or the duration of its action, maintaining in this manner the distance by which the particles are displaced under the action of the field at the previous level. Measurements can be repeated every 2 to 3 minutes, since the particles are able to return to their original position even when the viscosity of the system under study is high. Figure 5 gives the results of measurement of the viscosity of epoxy resin in the process of hardening (the volume concentration of the hardener is 10%) at different temperatures. A stable response of the optical signal and reliable results are observed up to the gelation point of the medium under study (these are time fluctuations in the level of the signal) throughout the temperature range re-



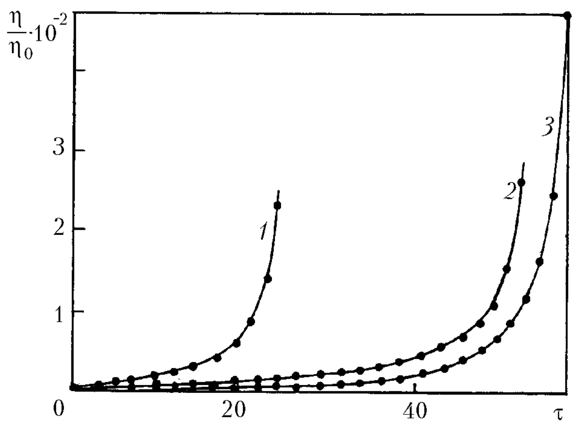


Fig. 5. Kinetics of viscosity of epoxy resin in the process of polymerization: 1) t = 50; 2) 70; 3) 100°C. η, P; τ, min.

Fig. 6. Change in the viscosity of a polymer film with drying time for different initial thicknesses of the film: 1) 100; 2) 200; 3) 225 μm. τ, min.

alized experimentally. The application of this method enables us to stably record a change from units to tens of thousands of poises in the dynamic viscosity of the oligomer composition in the process of its hardening.

Measurement of Rheokinetics in the Process of Drying of a Polymer Film. We have investigated the kinetics of viscosity of a polymer film formed into a solid state by evaporation of the solvent from a fairly low-viscosity solution of polyvinyl butyral in ethyl alcohol. The solution (1:5) was prepared by 12-h swelling at room temperature and 7-h boiling. The results of measurement of the kinetics of the medium's viscosity at room temperature are presented in Fig. 6.

The above examples demonstrate the great possibilities of the method developed for investigation of the viscosity of different fluid materials and their rheokinetics, both manifesting itself due to the physicochemical processes in the liquid itself and induced by external actions.

The advantages of the method are the small dimensions of the measuring cell and volumes (10^{-2} cm^3) of the medium under study and the simplicity and rapidity of measurement. There is a possibility of thermostatting, sealing hermetically, or insulating the specimen under study from the action of the external medium or, conversely, carrying out measurements under the action of physical fields of different nature on the liquid under study.

NOTATION

s, degree of filling of the layer; H, field intensity; t, temperature; β, slope of the curve of optical response; τ, time; η, viscosity; η_0, viscosity at a temperature of 20°C.

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