Rheology of Surface Films. III. A New Type of Surface Rheometer using Electromagnetic Driving Technique[1]

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Introduction

In the course of our effort to establish the standard set of surface rheometry, which involves the rheological measurements of films at liquid surface, the author has previously presented a surface rheometer of wire ring type[2] which enables the film creep to be measured under constant shear stress by using a phototube relay system. This apparatus, although it showed considerable merit in facilitating quantitative analysis of the creep data, was somewhat unsatisfactory in practice, since the construction was comparatively complicated and the operation was not so easy. In addition, it was not readily accessible to the measurements other than the creep test, that is, the stress relaxation test or the dynamic measurement. In order to eliminate these objections and permit wide application to various sorts of measurement, the author again originated a new type of apparatus, using electromagnetic driving technique. The advantage of this equipment lies not only in its ease of operation and simplicity of construction, but also in its accessibility to both static and dynamic measurements. Moreover, the remote control of the instrument, being readily capable using a pair of wires, greatly reduced the experimental care to provide vibration-free operation. The present paper describes the construction of this surface rheometer for static measurement while the description for dynamic measurement will be made in the near future.

Construction of Apparatus

The general view of this apparatus is shown in Fig. 1. Principal construction bears resemblance

1) Presented at the Symposium on High Polymer Chemistry of the Chemical Society of Japan, Oct., 1953, Tokyo.

2) K. Inokuchi, This Bulletin, **27**, 203 (1954).

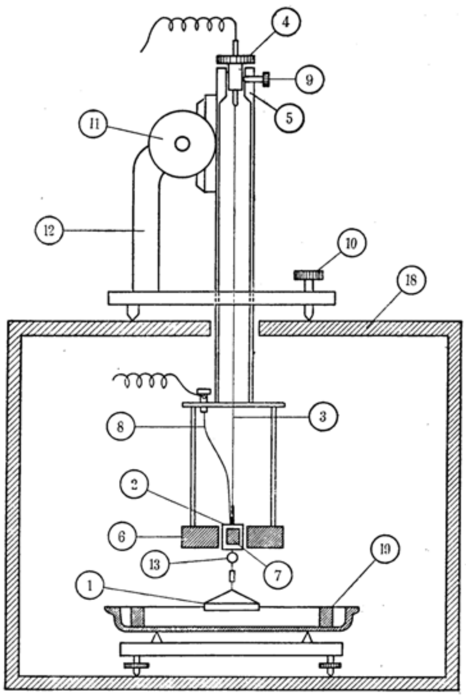

Fig. 1. Elevation view of the instrument.

to the d'Arsonval galvanometer. The actual suspension consists of a ring (1), a moving coil (2), a suspension wire (3), a support rod (4), and a guide (5). The moving coil is just situated between the poles of the permanent magnet (6). The ring is fixed to the moving coil through a small chuck. A light rectangular moving coil is suspended so that its sides lie in the air gaps between the two poles of a permanent magnet and a soft-iron cylinder (7) inside the poles. The suspension wire is an extremely thin strip of copper, which also serves as one of the leads to the coil, the other lead being a loosely hanging thin wire (8) leading upwards from the top of the coil. When the current passes through the coil, a deflecting torque is produced owing to the reaction between the permanent magnetic field and the magnetic field of the coil. Since the pole faces of the permanent magnet are shaped so as to give a radial field and the air gaps between the magnetic poles and iron cylinder are small, the flux density becomes uniform in a radial direction. Hence, the torque causing the coil to rotate turns out to be proportional to the current and constant for all positions of the moving coil as long as its sides are within the pole arcs of the magnet. The magnitude of the torque, therefore, can readily be known by the current of the coil multiplied by a constant.

The level of the coil can be adjusted by raising or lowering the support rod, which is held in place by the screws (9) during run. The centering of the coil is also adjusted by the levelling screws (10) of the stand (12). When the instrument is out of measurement, the support rod is lowered so that the coil becomes mounted on the iron cylinder (7), setting the suspension wire relaxed. The suspension carries a small mirror (13) below the coil upon which a light beam is cast. The beam of light is reflected onto a scale or to a recording photographic paper upon which the deflection is recorded.

The current is supplied from the potentiometer (14), the connection of which is illustrated in Fig. 2. A micro-ammeter (15) is inserted into the circuit in series with the moving coil (2). In order to avoid the effect due to the eddy current which appears in the coil of the ammeter (15) at the instant of closing the circuit, the coil (2) of the ring provides a resistance (16) parallel with it, the magnitude of which is adjusted to be the same as that of the coil (2). The changeable switch (17) offers the alternative connections of the circuit either to the resistance (16) or to the moving coil (2), the operation of which will briefly be described later.

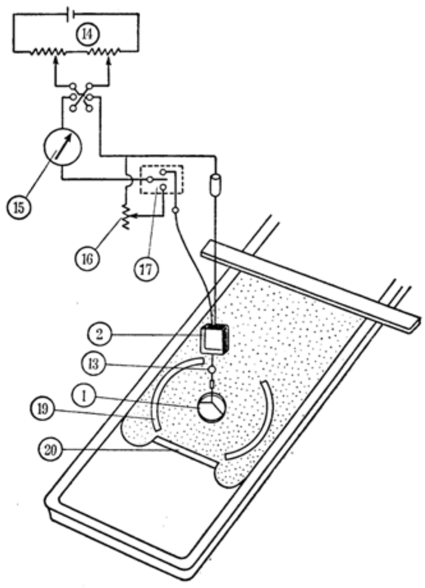

Fig. 2. Schematic illustration of the equipment.

The ring is a shallow cylinder of platinum, the upper half of which is treated with paraffin, whereas the lower half is left unconditioned. The size of the ring is 3.0 cm. in diameter and 0.3 cm. in height. The moment of inertia of the suspending system was 1.12 g. cm^2. The torsion constant of the suspending wire was 0.016 dyne. cm.

The stand (12) of the instrument was laid on the cabinet (18), only the suspending system being enclosed in it. The instrument was so set that the ring was placed towards one end of the trough, the size of which was $60.0\times15.0\times10.0$ cm. The ring was provided with a guard (19) of a pair of arc, which was placed in the trough concentric to the ring. The top of the guard was just the same height as the brim of the trough and coated the top face with paraffin for water-repellency.

Determination of Instrumental Constant

The determination of the constant relating the current passed through the moving coil (2) to the deflecting torque applied to it was made as follows. The torsion wire, of which the torsion constant was previously determined, was attached to the moving coil instead of to the ring, and the lower end of the wire was fixed to the ground. The knob (11) was carefully levelled so as to put the wire in proper tension between the coil and the ground. The varying amount of current was let into the coil from the potentiometer, and the respective deflection of the coil was read on the scale by means of a lamp and scale system. As shown in Fig. 3, linear plots were obtained in the cur-

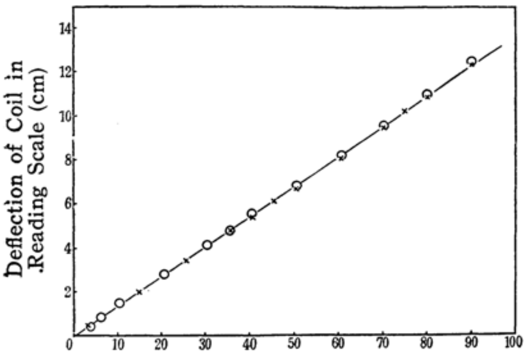

Fig. 3. Current-deflection curve of the moving coil. Circles and crosses represent the plots belonging to different run of measurement.

rent-deflection curve, showing the following equation to be valid

$$\theta = L \cdot m \tag{1}$$

where θ is the deflecting angle, m, the current, and L, a constant. Since the deflecting torque,* T, is directly proportional to the deflecting angle θ, the following equation holds

$$T = k \cdot \theta \tag{2}$$

where k is the torsion constant of the wire attached below the coil. Hence it turns out from the above equations that the torque is proportional to the current as seen in the following equation

$$T = (L \cdot k)m = p \cdot m \tag{3}$$

where p, being equal to $L \cdot k$, is a constant relating the current to the deflecting torque. In the present apparatus, p was calculated to 0.115 dyne. cm., when m was expressed in microammeter.

Procedure of Operation

The surface of water was cleaned by sweeping with moving barriers. After the ring was lowered so as just to touch the surface of water, the material under test was spread on the surface of the water. By raising carefully the support rod (4) and adjusting the levelling screws (10) for centering the coil, the ring (1) was allowed to be free in suspention. After adequate time was allowed for the equilibrium to be attained, the current was applied to the moving coil (2) of the ring. Prior to the measurement, the current was sent into the circuit turning the switch (17) so that the circuit was closed through the resistance (16), not through the coil (2). In this instant, only the ammeter was actuated by the amount of current which would be applied to the coil in case of measurement. At the time of measurment, the switch (17) was changed to the opposite side so that the current was sent into the coil (2), and the torque was now supplied to the ring without being affected by the eddy current due to the motion of the coil of the ammeter (15).

The deflection of the ring was measured on the scale, or if necessary, recorded on the photographic paper of the rotating drum. Since the deflecting torque of the ring is constant irrespective of the position of the ring if only the current passed through the coil is held constant, the creep curve under constant shear stress can readily be obtained under a constant current throughout the experiment.

If the potentiometer is so adjusted that the deflection of the ring is fixed to a certain value throughout the experiment, and the respective current is measured with time, the stress relaxation curve can readily be obtained.

In calculating the physical constants for the surface viscoelasticity, the film was considered as a mathematical plane. The surface shear modulus of rigidity (G) and surface viscosity coefficient (η) were obtained as follows, respectively, provided that the torque due to the moment of inertia of the rotating system could be ignored,

$$G = \frac{1}{4\pi}\left(\frac{1}{r_1^2} - \frac{1}{r_2^2}\right)\frac{p \cdot m}{\theta} \tag{4}$$

$$\eta = \frac{1}{4\pi}\left(\frac{1}{r_1^2} - \frac{1}{r_2^2}\right)\frac{p \cdot m}{\dot{\theta}} \tag{5}$$

where r_1 is the outer radius of the ring, and r_2 the inner radius of the guard coaxial to the ring, respectively.

In the present experimental condition, the stress as well as the strain applied to the film was not uniform throughout the film but varied with the distance from the center of the ring. The stress, S, applied to the outer radius of the ring was given by the following equation, when the moment of inertia of the ring might be ignored.

$$S = \frac{p \cdot m}{2\pi r_1^2} \tag{6}$$

In our usual experimental condition, in which the film resistance is sufficiently large compared with that of water, the water effect is negligibly small, and the immersion depth of the ring does not matter. However, if the film resistance is so small as to be comparable to that of pure water, the immersion depth would have to be taken into account.

* Since the torsion constant of the suspending wire was suffiiciently small compared with that of the wire attached below the coil, proportional constant between T and θ was simply taken as k, neglecting the deflecting torque due to the suspension wire.

Discussion

Strictly speaking, the deflecting torque, T, should be expressed as follows, taking the torque due to the moment of inertia of the rotating system into consideration

$$T = p \cdot m - I\ddot{\theta} \qquad (7)$$

where I represents the moment of inertia, and $\ddot{\theta}$, the acceleration of the motion of the ring. The author, however, expressed the deflecting torque, T, simply as $p{\cdot}m$ in equation (4) and (5), neglecting the term $I\ddot{\theta}$. This was because of the fact that, in our usual experiment, $I\ddot{\theta}$ was sufficiently small compared with $p{\cdot}m$, since very slow creep was mainly subjected to our experiment and the value of I is comparatively small. However, if it were the case, in which $I\ddot{\theta}$ could not be neglected against $p{\cdot}m$, the correction for the moment of inertia should, of course, be made.

The electromagnetic driving technique very much extended the range of measurement with satisfactory precision. The upper limit of the stress available might be decided by the permissible strength of the current through the wire, in order that over-heating might not occur through the coil. On the other hand, the lower value of the stress might be limited by the extent, to which the stress applied by the current can practically cover the second ary torque due to the torsional drag of the suspension wire, or to the asymmetry of the rotating system, since the present apparatus must satisfy the condition that the deflecting torque is independent of the position of the coil. Using a changeable shunt for the micro-ammeter, the apparatus permitted the measurement over a range of stress from about 3×10^{-3} to about 1×10^{1} dyne per cm. without changing any element of the coil. Such an extremely wide application of the stress is a practical advantage over the previous instrument using a torsion wire, in which a fairly large number of wires must be interchanged in order to apply such a wide range of stress.

The standard deviation of a set of data which appeared in the deflection-current curves was consistently less than 2% and the error inherent to the apparatus was considered to be practically satisfactory. The absolute values of visicosity or elasticity obtained with a different run of experiment using real films however, were found to fluctuate to a considerable extent. This may be due to some uncontrollable experimental conditions which affect the formation of the structure of solid films, and is, of course, not an inherent fault of the apparatus.

Another advantage of this equipment lies in the remote control of operation, which is readily available using a pair of wires. The remote control increases not only the ease of operation, but also serves to provide the vibration-free experimental condition, which is essential for the study of surface films.

In addition to the static measurement, the determination and the analysis of the dynamic modulus is also an important problem of the study. If the alternating current of proper frequency would be introduced into the coil of this apparatus, and the response of the ring would be detected with reference to the input stress, the present apparatus would also be accessible to the dynamic measurement of the film.

Summary

In order to establish the standard set of surface rheometry, which involves the rheological measurement of films at liquid surface, a new type of surface rheometer was devised using electromagnetic driving technique. The advantage of this apparatus lies not only in its ease of operation and simplicity of construction, but also in its accessibility to various sorts of measurement. In addition, the wide range of measurement and remote control of operation provide a practical advantage for the experiment of surface films.

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