A New Inductive Detection for the Magnetoviscometer

W. HERMANN and R. SOBCZAK, *J. Kepler University Linz, Department of Physical Chemistry, A-4020 Linz, Austria*

Synopsis

For a new magnetoviscometer with an inhomogeneous magnetic field to move iron spheres similar to the Stokes experiment, a nonoptical detection was developed. As a measuring principle the change of the impedance of a coil, penetrated by the sphere is used. The measuring principle is the change of impedance of an induction coil wrapped around the cell by the sphere. The measuring block, consisting of heating elements, detection coils, and a measuring cell is quite simple and therefore well suited for measuring under pressure. Since the proper cells are simply brass cylinders closed by a screw, they can be thrown away after the experiment (e.g., for network-building polymers). Examples for said measurements are given.

INTRODUCTION

A new viscometer using an inhomogeneous magnet field instead of the gravitational field usually applied in the case of Stokes's falling sphere experiment has been developed and applied successfully.[1-3]

In the present stage of development the force of the magnetic field equals about 150 times the force due to gravitation. This can be used to shorten the measuring time to look at very high viscosities (10^{11} Pa s at present) and possibly, but not tried yet, to produce high shear rates.

With the setup mentioned in Refs. 1, 2, and 3, we have investigated transparent polymers only. For this purpose we used a laser optical device (Fig. 1).

The laser beam is interrupted by the moving iron sphere which results in a signal at the photosensor. The velocity of the sphere (according to Stokes's law)

$$v_s = \frac{F}{3\pi\eta d}$$

where F (N) = force on the sphere, v_s (m/s) stationary velocity, d (m) = diameter of the sphere, and η (Pa s) = viscosity, has to be measured as exactly as possible. For that purpose laser and photosensor are mounted on a precision drive.

Since Stokes's law holds exactly for fluids of infinite extension only, one has to apply corrections for the cell geometry in practical cases. They were taken into consideration, of course and can be looked up for details in Ref. 2.

When using an optical method, we have to have windows which restrict the range of applicability considerably, since technical polymers especially with different kinds of additives are not transparent in general. Another problem

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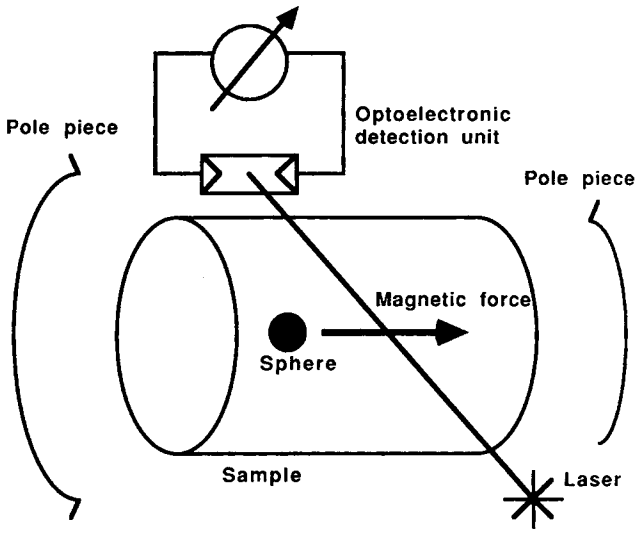

Fig. 1. Optical measuring principle of the magnetoviscometer.

which arises with windows is that they might not be tight enough and even break under higher pressures.

For us, however, there was still another reason for the development of another kind of detection. We had to do with substances containing small amounts of solvent. Heating generated a lot of small bubbles scattering the laser beam, and consequently an accurate measurement of the velocity was no longer possible.

To circumvent all these difficulties we developed another method to follow the movement of the sphere, the most important requirement being the absence of windows. A measuring principle which works even through the walls of a cell, provided that it is not built of ferromagnetic material, is the change of impedance of an induction coil wrapped around the cylindrical cell by a moving iron sphere. The details will be described in the next part.

EXPERIMENTAL

The cylindrical measuring cell is surrounded by two separated coils. A moving sphere has the same influence on the inductivity of the coils as a movable iron core would have. Depending on the position of the sphere, the inductivity of the arrangement changes.

In our case with rather flat coils, we use the following approximation[4]:

$$\text{inductivity } L = 2.1 \times 10^{-6} N^2 r \left(\frac{r}{l + h} \right)^{3/4}$$

where N = number of windings, $r = (r_i + r_0)/2$ = average radius of the coil, $h = r_0 - r_i$ = height of windings, and l = length of the coil. Since μ_{Fe} is much bigger than μ_{air}, a comparatively small sphere alters the inductivity considerably. The working principle of this method is shown in Figure 2. The coils are fed by an ac current with a frequency of 1 kHz.

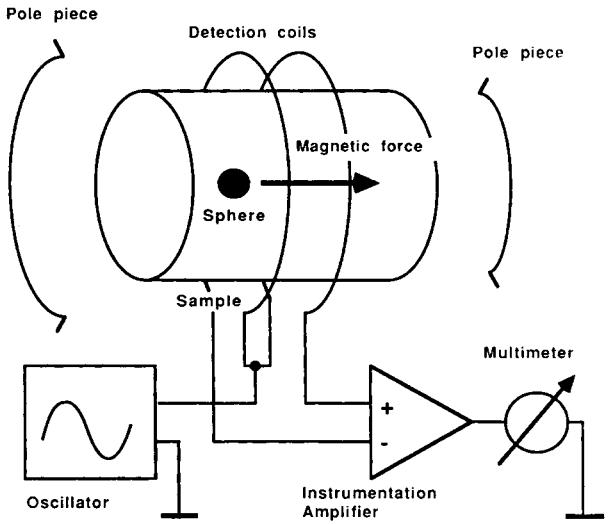

Fig. 2. Inductive measuring principle of the magnetoviscometer.

Changing the impedance of one coil by the penetrating sphere causes an imbalance since the impedance of the other coil, both being connected parallel to the ac source, is much smaller without the sphere in it. As can be seen in Figure 3, the respective currents are rectified and converted into proportional voltages: these are compared, and the resulting difference is amplified.

In Figure 4 the measuring block is presented as a whole. Two heating elements in cylindrical borings provide the temperature necessary. The space between the outer walls of the block and the inner cylinder (with the coils) is filled with silicon oil to get a homogeneous temperature distribution. The oil can be filled in by a tube which also serves as an inlet for a thermocouple.

The proper measuring cell is inserted into the inner cylinder of the block. It is very simply a brass (eventually other material) cylinder closed by a screw. Therefore, it is very cheap and can be thrown away after the experiment (in

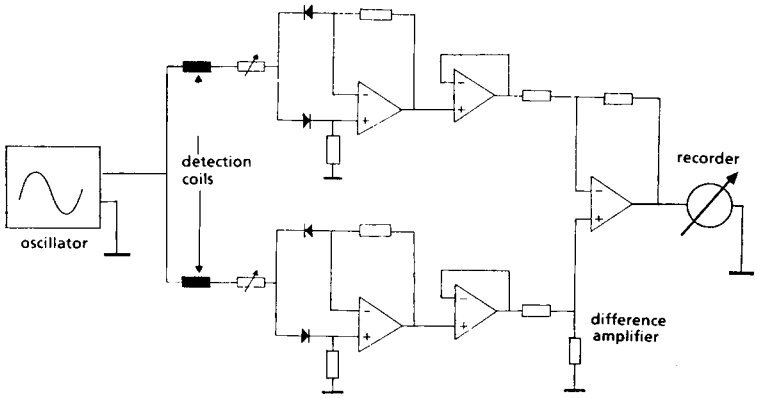

Fig. 3. Circuit diagram of the inductive detection.

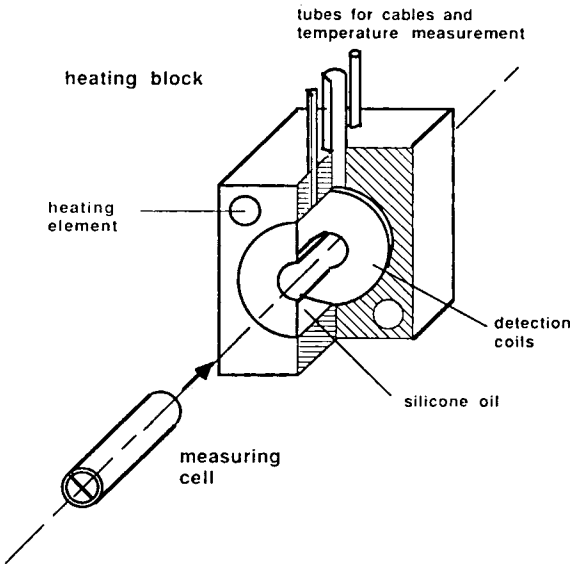

Fig. 4. Measuring block.

the case of epoxy resins which are rather insoluble, for example). The simple system is well suited for measuring under pressure as well.

In that case a cap which fits to a high pressure tube is used. Problems in connection with moving parts occurring in the case of rotation viscometers, or breaking windows using optical detection are avoided.

Our arrangement yields signals which depend on the size (mass) of the spheres. For instance, $d = 1$ mm, $U = 3$ mV, $d = 5$ mm, and $U = 200$ mV.

Figure 5 shows the signal of a sphere with $d = 3.5$ mm depending on its position. As mentioned briefly above, already this signal is the difference of the response of the separate coils. A part of the curve between its maximum and minimum is used as a scaling function for the position of the sphere (and in connection with time for its velocity). There is a definite correlation between the height of the signal (mV) and the position of the sphere (mm). The distance of the two extrema in the x-axis is 5.5 mm for the sphere described above.

This measuring distance is very well suited for lower viscosities like $\eta = 10$ Pa s. It takes a 1 mm sphere 1.04 s in a magnetic field ($I_M = 6$ A, force on the sphere $F = 0.0005$ N) to move the whole distance (5.5 mm). This time can be recorded easily. Even along that distance, the magnetic field (and force on the sphere) changes about 10% only. If this has to be avoided, a smaller measuring distance has to be chosen, which has to be done for higher viscosities ($\sim 10^{-2}$ mm for 10^{10} Pa s). On the other hand, with an average value of the force along the measuring distance, satisfactory results are yielded. The slope of $U(x)$ is biggest between the inner views of the two coils, and the sensitivity has its maximum there. This region is suited for the measurement of much higher viscosities. A 3.5 mm sphere needs 70 h in a melt of $\eta = 10^9$ Pa s to move just 0.1 mm.

The examples show that with the existing setup velocities varying by several orders of magnitude can be detected, and, therefore, a wide viscosity

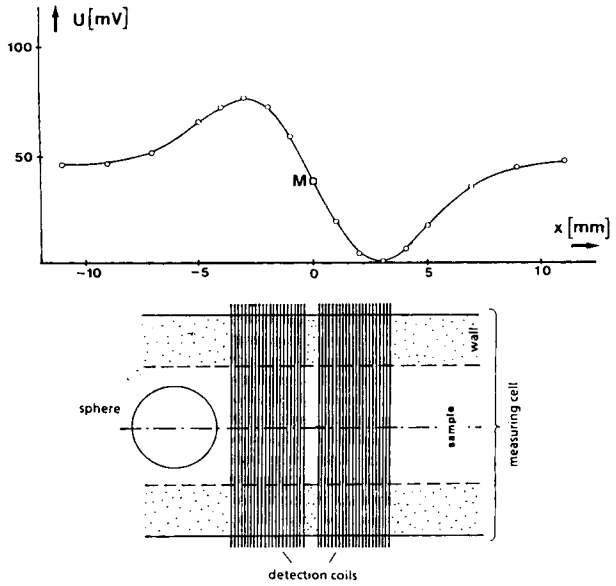

Fig. 5. Signal of the sphere (d = 3.5 mm) according to its position x in the cell.

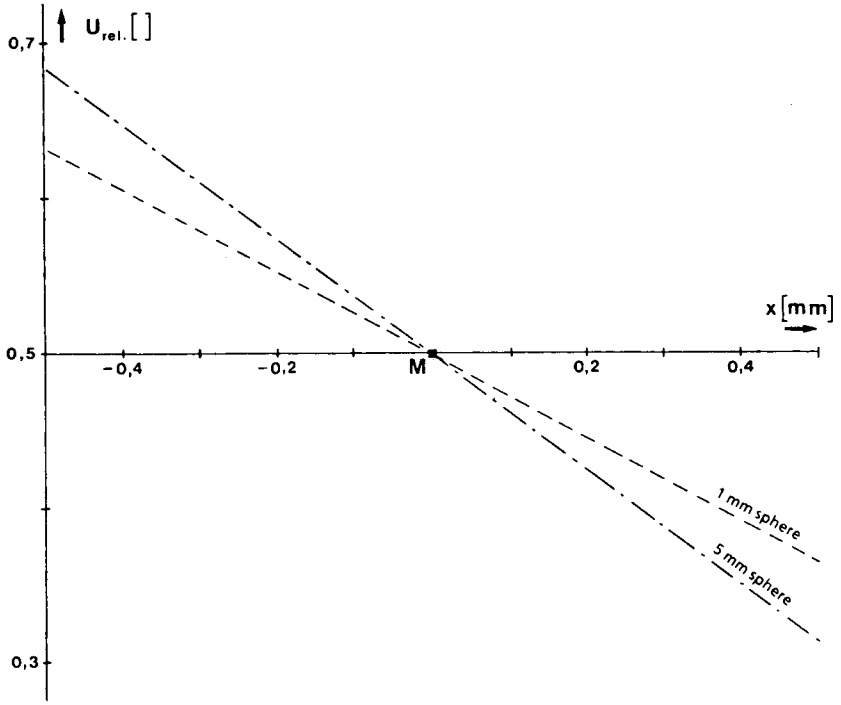

Fig. 6. Comparison of the relative signals U_{rel}.

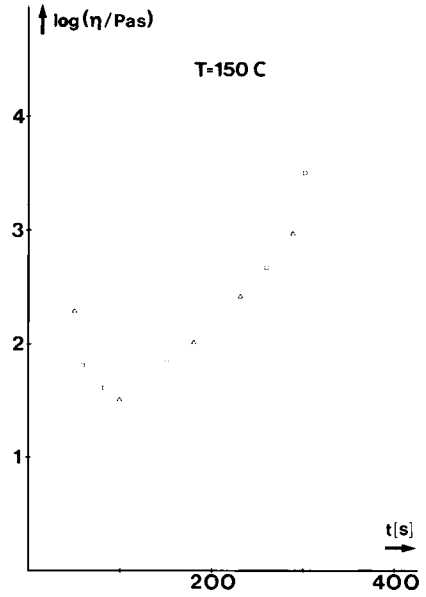

Fig. 7. Viscosity of a curing epoxy resin.

range is covered. This is especially important looking at systems with viscosities changing fast in a wide range, as is the case in the buildup of polymer networks (polyester, epoxy resins). The present system has been chosen as a compromise, of course, to follow the time dependence of viscosity in epoxy resins mentioned several times.

This can be seen in Figure 6. The signal per mm on the x-axis of the 1 mm sphere is bigger than in the case of the 5 mm sphere. The reason is that the bigger sphere penetrates one coil already without having left the other one completely. To get a better resolution for bigger spheres also and a wider range toward lower viscosities, one might choose a larger distance between the coils.

RESULTS

1. The resin is rather insoluble after the buildup of the network. Therefore, the powdery samples (prepregs) are pressed into the throwaway cell with the chosen sphere, and pushed into the heating block which has been warmed up already. The block can be turned around by an angle of 180°, and the sphere can move along the measuring distance several times. This is necessary in the early stages of the reaction with its small viscosities and therefore higher velocities. The sample shrinks during the melting process; to avoid voids, the screws closing the cells can be tightened (by turning a bit).

As a characteristic example, a viscosity vs. time curve is shown for an epoxy resin (Multicon Electronic) at 150°C in Figure 7. At $t = 0$ the cell is inserted into the heating block. After the insertion of the cell into the heating block, the time to reach the measuring temperature in the center of the cell is about 90 s. Therefore, the first part of Figure 7 shows the decrease of viscosity of a

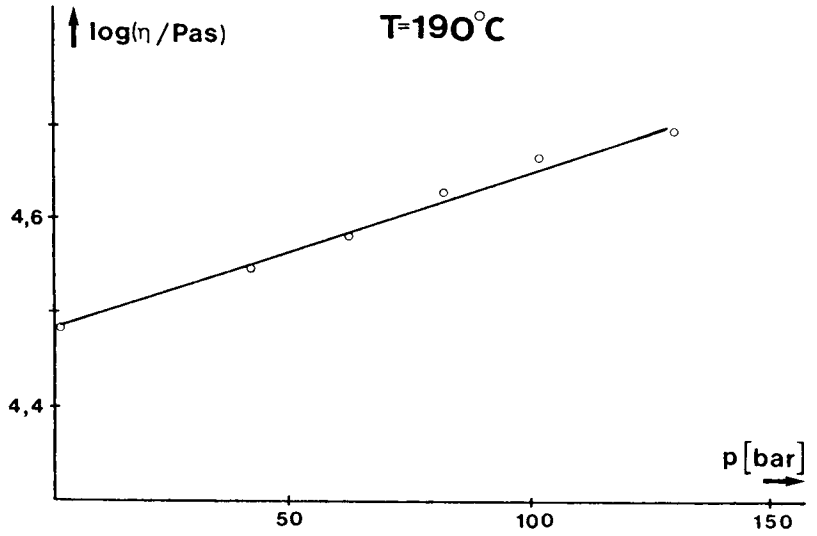

Fig. 8. Pressure dependence of the viscosity of polystyrene.

liquid with increasing temperature while the second part describes the rise of viscosity according to the growing mole mass of the resin.

Unfortunately, this method too does not allow us to determine the point of gelation. The highest values in the log η scale are certainly above that point. What is actually measured is the resistance of the network towards the movement of the sphere. With additional methods like DSC it should be possible to get some final conclusions about the gel point. The purpose of this contribution is rather to show that, with the method described, viscosities changing rapidly within a wide range can be followed with a comparatively simple setup.

2. Measurements under Pressure. For that purpose, cells built of nonmagnetic steel are used instead of brass cylinders. They are connected to an N_2 bottle via a high pressure tubing. The pressure is measured with the manometer of the bottle. With that arrangement the pressure dependence of the viscosity of a polystyrene melt (PS 165 H Chemie Linz) was measured as an example (Fig. 8).

We tried to describe the dependence using a formula according to Ramsteiner[5]

$$\frac{\eta_p}{\eta_{p_0}} = e^{\alpha p}$$

where η_{p_0}, η_p = viscosities at atmosphere and higher pressure, α = pressure coefficient, and p = pressure. In Table I[5] data of polystyrene of several mol masses and at different temperatures reported by several authors together with our own results are compiled. The correlation is satisfactory.

TABLE I
Pressure Coefficients of Different Polystyrenes

Mol mass × 10^3 (g/mol)	$\alpha \times 10^{-3}$ (bar^{-1}) T = 170°C	T = 190°C
7.5	4.82	
20		
25	5.88	
74	6.93	
196		4.8
267	4.32	
280	3.9	3.9
417	3.8	4.6
PS 165 H		4.3

CONCLUSIONS

The development of the induction (nonoptical) detection yields a more versatile magnetoviscosimeter according to the following points:

Viscosity Measurements of Nontransparent Substances

A measuring cell as simple as a tube which can be closed by a screw and thrown away in the case of unsoluble samples is used. These tubes can be machined in various sizes. They may be even very small if the sample available is small. Cells as tiny as 50 mm^3 were made and tested successfully.

Viscosity Measurements under Pressure

Since the proper cell has neither windows (for optical detection) nor rotating (moving) parts which have to be tight, the system is very well suited for measurements under high pressure. With the help of hydraulics and/or a compressor higher pressures should be accessible.

Another possibility of the inductive principle is to use, instead of the influence of the moving sphere on the impedance of the coil, the temperature dependence of the magnetic properties of Heusler alloys which has a similar impact. Thus the temperature in the sample can be measured without the insertion of any element (thermocouple) into it.

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