A PARALLEL PLATE RHEOMETER FOR MEASURING THE VISCOELASTIC PROPERTIES OF WHEAT FLOUR DOUGHS

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ABSTRACT

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A parallel plate rheometer suited to measuring various rheological functions is described. This instrument uses a linear air-bearing and maintains constant spacing between the plates to ensure that the sample deformation is simple shear. The ancillary measuring equipment can be changed without alteration to the sample cell so that the instrument can easily be adapted for other rheological measurements, for example, stress relaxation and dynamic measurements. Some results of creep and creep recovery measurements on doughs are reported to illustrate the use of the instrument.

When the strain and the stress are not uniform throughout a sample of a nonlinear viscoelastic material such as wheat flour dough, the rheological properties vary through the sample. The determination of the dependence of the mechanical properties on the strain amplitude from the experimental observations is then extremely difficult (1) as it is not possible to calculate the strain distribution through the sample unless the dependence of the rheological properties on the strain is already known. For the study of nonlinear materials it is, therefore, desirable to use a rheometer with a sample geometry and type of deformation which ensure that the strain is uniform throughout the sample.

Most of the instruments that have been used for measuring the mechanical properties of soft solids involve a deformation such that the stress and strain vary with position in the sample. These instruments are therefore not suitable for the study of nonlinear viscoelastic materials. The geometries and types of deformation which give homogeneous strain have been discussed in a recent review of the measurement of the fundamental rheological properties of wheat flour doughs (2). Only two sample geometries suited to making measurements on soft solids, such as dough, meet exactly the requirement of uniform strain: simple shear between parallel plates, and simple extension. The requirement is approximately met by the cone and plate geometry if the angle between the cone and the plate is small.

Many measurements have been made in extension using instruments based on the mercury-bath extensometer originally designed by Schofield and Scott Blair (3). With this method there are difficulties in achieving uniform stress close to the ends of the sample where the stress is applied. Thus, measurements of the change in length need to be restricted to the central portion of the sample where the stress is uniform. It is difficult to devise a simple recording system to monitor such changes in length.

Some measurements using the cone and plate geometry have been reported (4,5) and others are expected from cereal laboratories that have Weissenberg rheogoniometers. There are problems in loading a cone and plate instrument with a dough since the sample has to be extremely thin, especially near the center where, in theory, the cone and plate touch.

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Creep and creep recovery measurements have been made on doughs using the parallel plate instrument described by Nikolaev and Beganskaya (6). In their instrument the spacing between the plates can vary while the sample is being strained and the deformation is not constrained to be simple shear.

In this paper, a parallel plate rheometer employing a linear air-bearing is described and some measurements of creep and creep recovery are reported.

THE RHEOMETER: PRINCIPLES AND APPLICATION

The deformation of a material in simple shear consists of the relative displacement of parallel planes called shearing planes. Each shearing plane moves rigidly along a straight line (line of shear) in its own plane. The lines of shear for all shearing planes are parallel and the separation of each pair of shearing planes is constant. The relative displacement of any pair of shearing planes divided by their separation has the same value for all pairs of shearing planes and is taken as a measure of the shear strain.

The deformation of a sample in simple shear between parallel plates is illustrated in Fig. 1. The spacing between the plates is constant and there is no change in the volume of the sample. The shear stress is given by the tangential force, F, applied to the movable plate, divided by the area, A, of the plate. The shear strain is the displacement, d, of the movable plate divided by the thickness, h, of the sample.

The sample cell of the rheometer is shown in perspective in Fig. 2a and a side elevation of the instrument, equipped to measure creep and creep recovery is shown in Fig. 2b. The sample, A, is held between the two plates, B and C (200 mm × 75 mm). The upper plate, B, is fixed to a hollow block, D, which is supported by the rigid superstructure, E. The height of the upper plate and hence the thickness of the sample can be varied by spacers on the columns supporting the superstructure. The lower plate, C, is attached to a length of aluminum Y-section extrusion, F, which is constrained to move only in a straight line at a fixed height by a linear air-track, G, made from aluminum extrusion of triangular cross-section (125 mm × 88 mm × 88 mm).

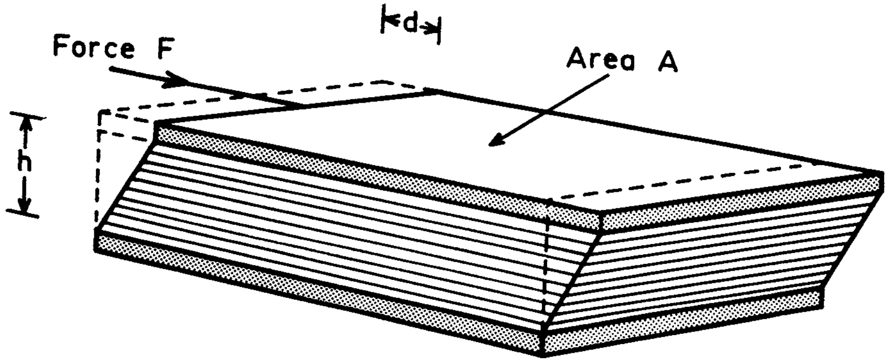

Fig. 1. Sample in simple shear between parallel plates. Stress = F/A; strain = d/h.

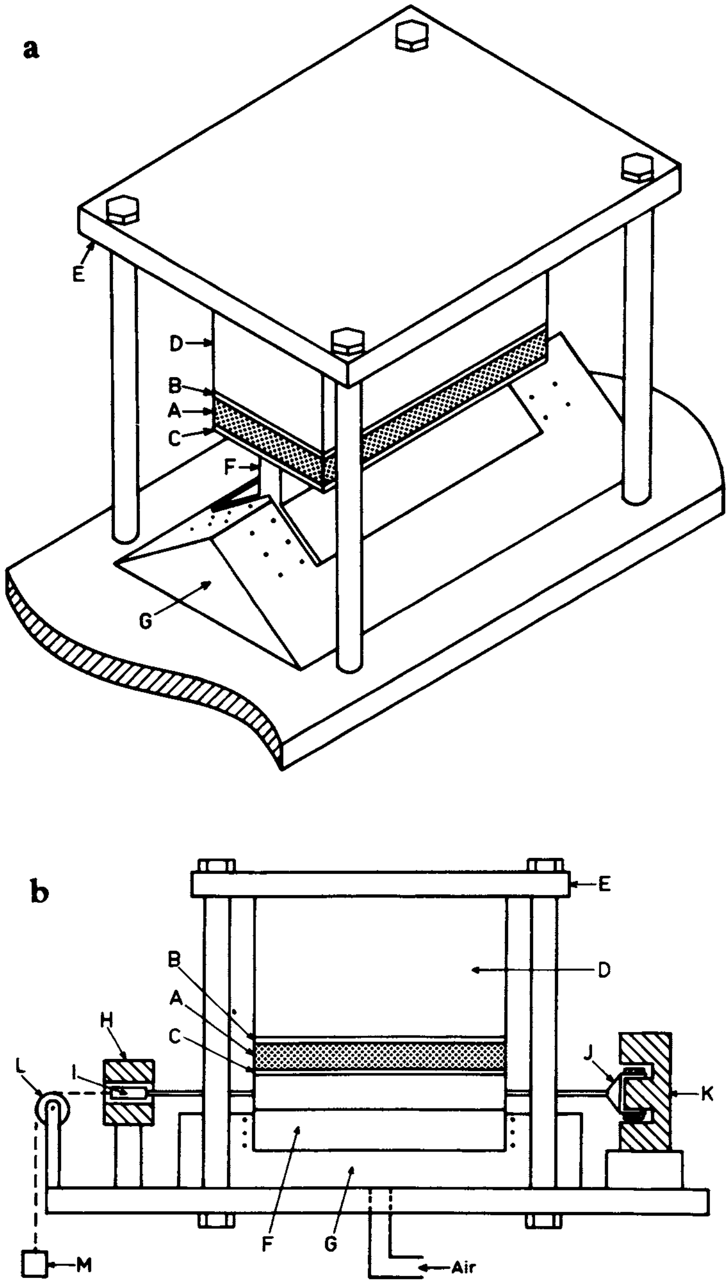

Fig. 2. Sample cell for creep and creep recovery measurements on dough in simple shear: a) perspective, b) side elevation. A, sample; B, fixed plate; C, movable plate; D, hollow block; E, rigid frame; F, sliding Y-section; G, air-track; H, differential transformer; I, ferromagnetic core; J, coil; K, permanent magnet; L, pulley; M, load.

Air is supplied, at a pressure of 2.5 kPa, to the inside of the air track and escapes through rows of 0.8 mm diameter holes with a grid spacing of 12 mm on the upper surfaces. The air bearing supports the movable slide and an additional load of up to 1 kg.

The upper plate, B, together with the block, D, and the upper part of the superstructure can be lifted to facilitate filling of the sample cell with dough. An excess of dough, rolled into a cylindrical shape, is placed on the central axis of the lower plate, C, and then the upper plate, B, is lowered, expelling the surplus dough which is trimmed off with a sharp scalpel. The exposed surface is immediately coated with soft petroleum jelly and painted with a hydrocarbon oil to prevent moisture loss from the sample. The exposed surface area depends on the separation and size of the plates. With most configurations the spacing between the plates is small compared with the other dimensions and the exposed area is small for the volume of the sample. When filling the sample cell, some flow of the dough occurs during expulsion of the surplus but the stresses involved will not be excessive unless the gap between the plates is very small. The temperature of the sample is controlled by circulating water through the block, D, and by controlling the temperature of the air supplied to the air-bearing. To eliminate errors arising from differential thermal expansion, the instrument is housed in a temperature controlled enclosure.

For measurements of creep and creep recovery, the displacement of the plate, C, is measured using a linear variable differential transformer, H, which measures the displacement from a reference point of a ferromagnetic core, I, attached to the movable assembly. The shearing force can be applied by a simple electromechanical transducer (7); the coil, J, attached to the movable assembly is in the radial field of a cylindrical permanent magnet, K. The loading pattern, a step function for creep, is obtained by passing a current through the coil. Alternatively, for higher loads the force may be applied by a thread passing over a pulley, L, to a freely hanging load, M.

The lowest stress which can be applied by mechanical loading is limited by the friction of the pulley to about 10 N/m^2. When using the electromechanical transducer the only resistance to movement of the lower plate, other than from the sample, comes from the fine coiled wires providing electrical connections to the coil and the friction of the air track. The frictional forces are extremely low and the limit to measurement has been the sensitivity of the circuits used to measure the displacement. It has been possible to make creep measurements down to a stress of 1.5 N/m^2.

EXPERIMENTAL

The creep and creep recovery measurements reported in this paper, to illustrate the use of the parallel plate rheometer, were all made from similar doughs mixed from the same batch of a commercially available Australian flour (protein 12.6%, moisture 13.0%, diastatic activity 195 mg maltose/10 g flour, Farinograph water absorption 65.2%, Farinograph development time 4.6 min). The doughs were prepared at 27°C by mixing 300 g of flour, 6 g of salt, and 186 g of water for 5 min in a Hobart mixer.

After loading into the sample cell, the sample was allowed to rest for 90 min to allow relaxation of the stresses induced during mixing and filling. This rest period also allowed the temperature of the sample to equilibrate to the selected control temperature of 27° C. The load was then applied for the selected time and the displacement of the lower plate was recorded for creep during the period the load was applied and for recovery after the load was removed. In all the experiments reported in this paper the load was applied at time zero and removed after 250 sec.

RESULTS AND DISCUSSION

All the creep and creep recovery curves are similar to those characteristic of a non-crosslinked polymer. Typical results are shown in Fig. 3 where the observed displacement of the movable plate is plotted against time for three levels of applied stress. The displacement does not approach a constant value and, therefore, there is no equilibrium compliance. The rate of displacement appears to have been approaching a constant value before the load was removed but this condition was not reached in the period for which the load was applied; the rate of displacement was still falling with time when the load was removed. After removal of the load, the sample recovers some of the deformation but does not return to its original condition because there remains a permanent deformation. This conclusion has been confirmed by observation of recovery over longer periods than shown in Fig. 3.

The design of the sample cell, the constraints on the relative motion of the two plates and the virtual incompressibility of unyeasted doughs ensures that all the requirements of simple shear are met provided that the sample adheres to the plates and that end effects associated with finite sample geometry may be

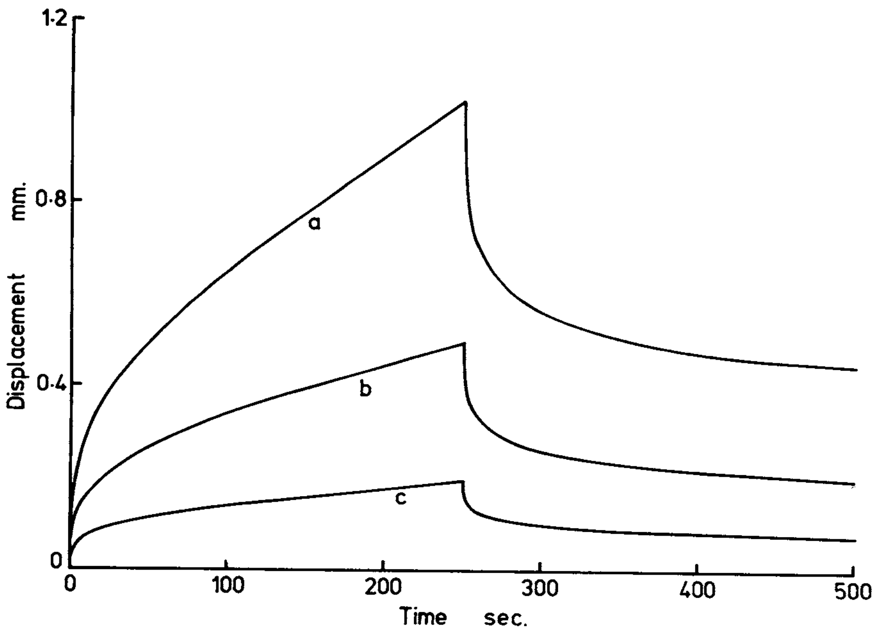

Fig. 3. Typical creep and creep recovery curves for three levels of applied stress: a) 100 N/m^2, b) 70 N/m^2, c) 30 N/m^2. Sample thickness = 5.0 mm.

neglected. If the deformation is simple shear then the displacement of the movable plate at any given time would be proportional to the sample thickness for a given loading pattern. Measurements were made on samples of various thicknesses to test whether the deformation conformed to this shearing pattern. In Fig. 4a the displacements of the lower plate observed at 10, 50, and 250 sec after the application of a stress of 100 N/m^2 are plotted against the sample thickness, and in Fig. 4b the recovered displacements, after the stress had been applied for 250 sec and then removed, are plotted for 10, 50, and 250 sec after the stress had been removed. Over this range of sample thickness (~1 − 8 mm) the sample is subject to simple shear and the shear strain must be uniform and the assumptions inherent in the method are valid.

Attempts to load the sample cell with thicker samples (> 8 mm) led to problems in trimming the sample. Because of the nature of the dough the sample pulled away from the edges of the plates. It was not practicable to load the cell with samples thinner than about 1 mm as the air track could not support the large forces required to force the surplus dough from between the plates. Such large forces are undesirable as they could change the character of the dough. The choice of a suitable sample thickness depends on the properties of the material being tested. For the dough used in these tests, a suitable sample thickness was about 5 mm and the following results were all obtained on samples of thickness 5.1 mm.

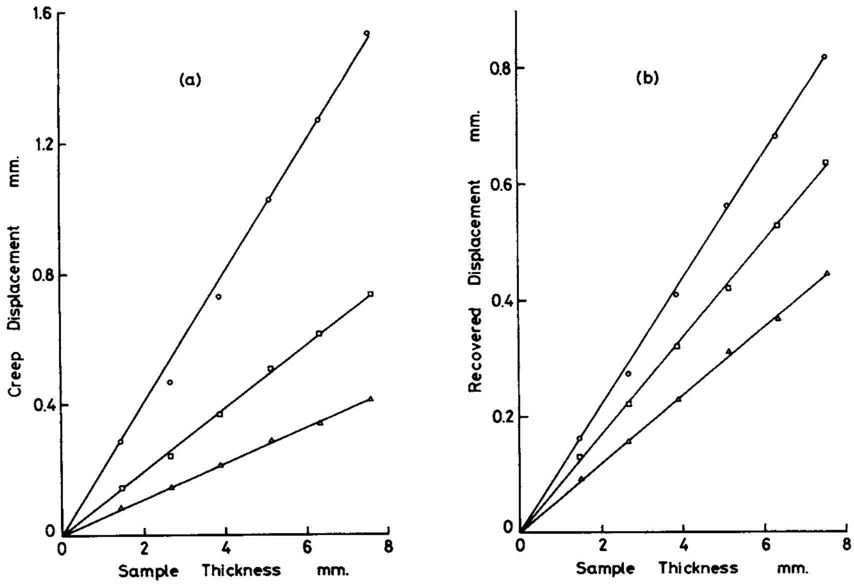

Fig. 4. a) Creep displacement for dough stressed at 100 N/m^2 and b) recovered displacement after load is removed plotted against thickness of sample. Points are plotted for three times after the start of creep or creep recovery: △ 10 sec, □ 50 sec, and o 250 sec.

The plots of creep strain against stress for various times after loading are shown in Fig. 5a. The recovered strains at various times after removing the stress are plotted against the stress in Fig. 5b. These plots would be straight lines passing through the origin for a linear viscoelastic material. Clearly, there is no truly linear viscoelastic region for this dough. However, the deviation from linearity is not greater than 10% for stresses up to 40 N/m^2 and the behavior of this dough may be considered to approximate to that of a linear viscoelastic material only over this range of stresses. At higher stresses the curves show a marked deviation from straight lines passing through the origin exemplifying the essential nonlinear character of dough.

The scatter of the experimental points from the curves in Fig. 5 is remarkably small as the points for each level of stress were obtained from different batches of dough. Thus the variations between samples, arising from the unavoidable differences in the mixing of the doughs and the filling of the sample cell, do not cause large changes in the properties of the dough after it has relaxed for 90 min.

Fig. 6 is a similar plot to Fig. 5 with the scales magnified so that the experimental points from measurements at low creep stresses can be shown in greater detail. These plots cover the range where the stress can be applied using a load and a pulley or by passing a current through the coil of the electromechanical transducer. The results are independent of the method of applying the stress. Lower stresses may be applied using the transducer than have been achieved with other instruments. Even at very low stresses the curves appear

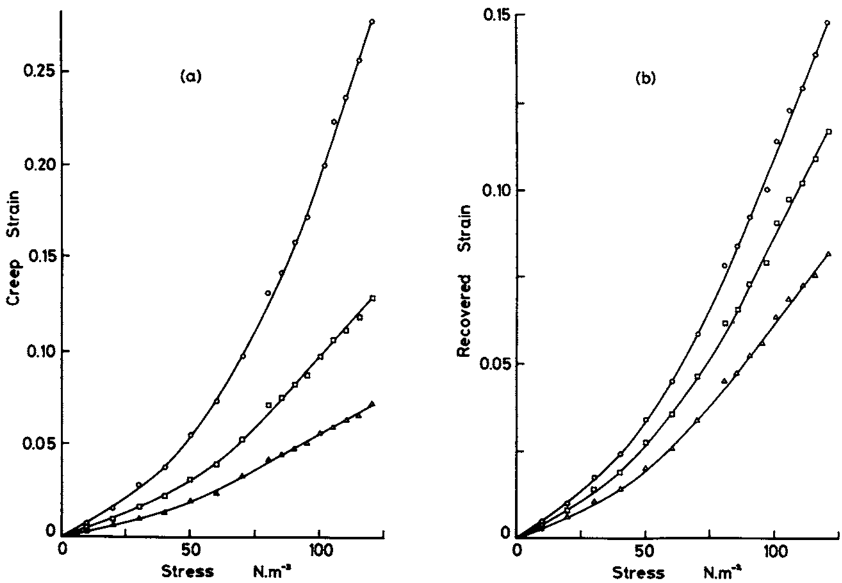

Fig. 5. a) Creep strain and b) recovered strain plotted against stress. Points are plotted for three times after the start of creep or creep recovery: △ 10 sec, □ 50 sec, and o 250 sec. Points determined using load and pulley.

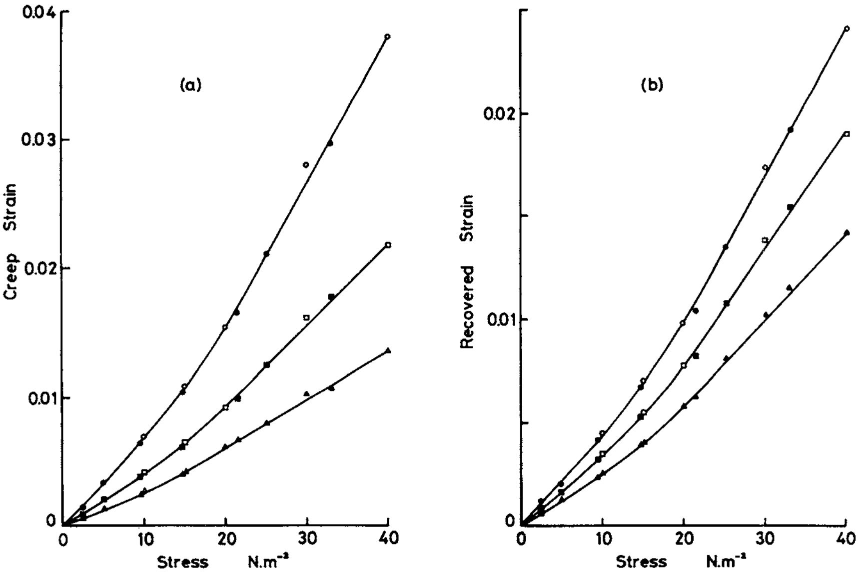

Fig. 6. a) Creep strain and b) recovered strain plotted against stress. Points are plotted for three times after the start of creep or creep recovery: △ 10 sec, □ 50 sec, and o 250 sec. Filled points determined using current through coil and open points using load and pulley.

to be continuous and to pass through the origin; there is no evidence for the existence of a yield value.

Previously reported studies of the creep of wheat flour doughs have been summarized by Hibberd and Parker (2). Comparison of the results given in this paper with other published results shows that the creep curves are qualitatively similar. However, there are differences between our observations and those of previous authors. Firstly, the results do not give any evidence for the existence of a yield value even though measurements have been made at much lower stresses (1.5 N/m^2) than the stress suggested by Bloksma (4) for a yield value (~15 N/m^2). Secondly, the viscoelastic behavior of dough is essentially nonlinear and the region over which the behavior is even approximately linear is limited to very low stresses and strains, well below the stresses and strains involved in manufacturing processes in industry.

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