Nonlinear Creep and Creep Recovery of Wheat-Flour Doughs

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ABSTRACT

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Measurements of creep and creep recovery of nonyeasted wheat-flour doughs have been made in simple shear over a range of applied stresses and with the stress applied for a range of times. The measurements confirm the nonlinear viscoelastic character of dough, give no evidence for a yield value, and show that within the time of measurement of creep (up to 10,000 sec) the rate of flow does not become constant, ie, the flow cannot be considered to become purely viscous. Over a limited range of stress and time, the nonlinear creep behavior can be characterized by independent functions of stress and time. These functions can be used to predict the recovery for short times after removal of the stress.

Measurement of creep compliance, ie, the strain divided by the stress as a function of time, in response to a stress that is applied suddenly and then maintained constant, is one of the standard methods of characterizing the mechanical properties of a viscoelastic material (Ferry 1961). For *linear* viscoelastic materials, the creep compliance is independent of the applied stress and completely defines the mechanical behavior. The response to other patterns of deforming forces can be predicted, using the Boltzmann superposition principle, provided the creep compliance is known over a sufficiently wide range of time. In particular, the stress generated in response to a step change of deformation (stress relaxation) and the response to a sinusoidally varying stress or deformation (dynamic behavior) can be calculated (Gross 1953).

The characterization of the mechanical behavior of *nonlinear* viscoelastic materials is more complicated because the nonlinear creep compliance is a function of both stress and time. Furthermore, even with a knowledge of this dependence on time and stress, the response to other loading patterns cannot be predicted without a constitutive relation between stress, strain, and time. The Boltzmann superposition principle cannot be applied.

Measurements of the creep behavior of doughs (Bloksma 1962, Glucklich and Shelef 1962a, Hibberd and Parker 1978, Matsumoto et al 1972, Nikolaev and Beganskaya 1954, Schofield and Scott Blair 1933, Smith and Tschoegl 1970, Yoneyama et al 1970) have been made using a wide range of instruments. Often it is not possible to distinguish between true dough behavior and instrumental artifact, particularly where it is claimed that "instantaneous" elasticity has been measured. The problem of comparing dough properties is compounded by variations arising from real differences between doughs prepared from a range of flour types using different compositions and mixing procedures.

In general, measurements of creep and creep recovery of doughs have covered only a limited range of experimental conditions. (Only Bloksma [1962] has discussed the results in terms of a stress-dependent compliance for measurements over a wide range of the applied stress.) Published results have been interpreted with a variety of inconsistent conclusions. The only commonly agreed conclusion is that dough is viscoelastic and behaves as a typical noncross-linked polymer. There is no agreement concerning whether dough has a yield value, whether its behavior at low stresses can be approximated by linear theory, or whether the flow rate becomes constant at long creep times.

In this investigation, creep and creep recovery were studied in simple shear. This type of deformation was chosen to ensure that the stress and the strain, and hence the mechanical properties of the dough, are uniform throughout the sample (Hibberd and Parker 1975). This is an essential condition in the experimental determination of the stress-dependence of the nonlinear compliance.

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EXPERIMENTAL

The creep and creep recovery measurements reported in this paper were made in simple shear using a parallel plate rheometer (Hibberd and Parker 1978). Two commercial Australian flours were used. In the first experimental series (constant creep time with various applied stresses) the flour had protein 12.7%, moisture 12.8%, diastatic activity 2.31 mg maltose/10 g flour, farinograph water absorption 64.8%, and farinograph development time 4.4 min. For the other series of experiments (different creep times at a single stress) the flour had protein 12.6%, moisture 13.0%, diastatic activity 1.95 mg maltose/10 g flour, farinograph water absorption 65.2%, and farinograph development time 4.6 min. The doughs were prepared at 27°C by mixing 300 g flour and 6 g of salt for 3 min in a Hobart mixer with 186 g of water for the first flour or 189 g of water for the second.

The experimental procedure was exactly as described previously (Hibberd and Parker 1978) with the sample allowed to rest for 90 min after loading before the stress was applied for the selected time. Each creep and creep recovery measurement was made on a new sample from a fresh mixing of dough.

RESULTS AND DISCUSSION

The creep and creep recovery curves were all qualitatively similar to previous measurements (Hibberd and Parker 1978) and to those published by most other authors (Glucklich and Shelef 1962a, Matsumoto et al 1972, Nikolaev and Beganskaya 1954, Schofield

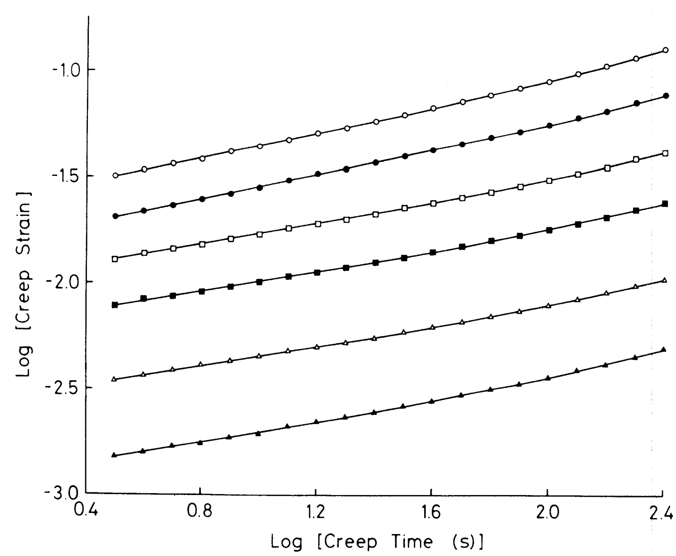

Fig. 1. Creep strain plotted against creep time for various stresses. ▲ = 10 N/m^2, △ = 20 N/m^2, □ = 40 N/m^2, ■ = 60 N/m^2, ● = 90 N/m^2, o = 120 N/m^2.

and Scott Blair 1933, Smith and Tschoegl 1970, Yoneyama et al 1970). However, there were no discontinuities of inflection points, as reported by Bloksma (1962), on any of the creep curves.

The creep strains for the first series of experiments, in which various stresses were applied for 250 sec, are plotted as a function of time in Fig. 1 using logarithmic scales. Curves for only six applied stresses are shown for clarity.

If dough were a linear viscoelastic material, these logarithmic curves would be superimposed by a vertical shift equal to the logarithm of the applied stress. The curves for all stresses, over the range 10 to 120 N/m^2, can be approximately superimposed by vertical shifts but the shifts are not equal to the logarithms of the applied stresses. The fit obtained by vertical shifting is better at low stresses and for short times than for higher stresses and longer times. The approximate superposition by vertical shifting suggests that, under certain conditions, the effects of stress and time may be separated. That is, the nonlinear creep compliance may be expressed as the product of two functions, viz., a time dependent compliance taken at an arbitrary reference stress, and a function of the stress that is independent of time. Both functions depend on the reference stress. Without loss of generality, the reference stress can be taken as zero so that we have:

$$\mathcal{J}(\sigma,t) = S(\sigma)J(t) \qquad (1)$$

where $\mathcal{J}(\sigma,t)$ is the nonlinear compliance at time, t, and stress, σ, $S(\sigma)$ is the function of the applied stress, and J(t) is the limiting value of the nonlinear compliance as the stress approaches zero.

TABLE 1
Ratio of Slope to Intercept of Straight Lines Fitted to Compliance vs. Stress Plots

Creep Time (sec)	A_1[a] (m^2/N)	A_2[b] (m^2/N)
3.2	0.0063	0.0084
6.3	0.0069	0.0085
10	0.0076	0.0087
16	0.0083	0.0087
25	0.0089	0.0087
40	0.0098	0.0087
63	0.0104	0.0086
100	0.0111	0.0086
158	0.0116	0.0084
250	0.0129	0.0086

[a] A_1 for lines fitted 10–120 N/m^2.
[b] A_2 for lines fitted 10–60 N/m^2.

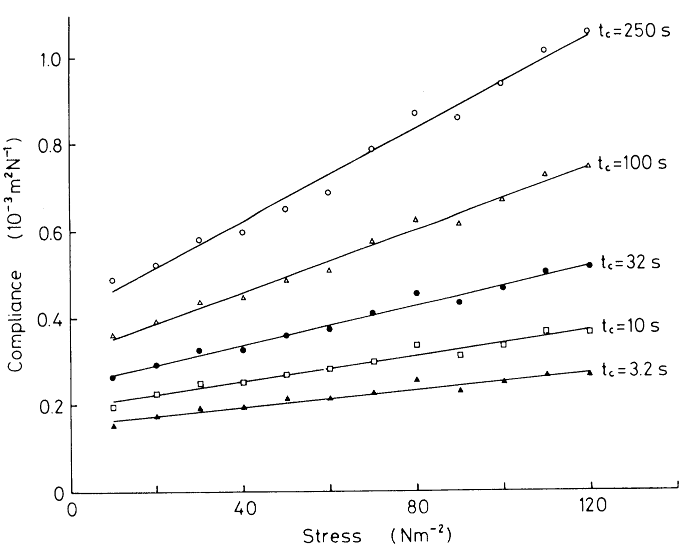

Fig. 2. Creep compliance plotted against applied stress at various creep times.

The function, J(t), cannot be measured directly but can be determined by extrapolating the ratio of the strain divided by the stress at low values to zero stress.

Plots of the compliance at selected times are plotted against the stress in Fig. 2. The well-defined systematic trends shown by the lines are remarkably good as each point for a given time is derived from a different mixing of dough. The variations arising from mixing and loading of the samples are relatively small. This contrasts with the scatter of points published by other authors.

The variation of compliance with stress shows a consistent trend over the whole range of experiments. A linear viscoelastic region requires that the compliance be constant over a range of stresses. It is clear, therefore, that for this dough there is no linear region at stresses above 10 N/m^2. Previous results (Hibberd and Parker 1978) have shown that there is no truly linear viscoelastic behavior at even lower stresses.

The straight lines of Fig. 2 suggest that for stresses within the range of these measurements (10–120 N/m^2), the creep may be represented by an expression of the form:

$$\mathcal{J}(\sigma,t) = [A\sigma + 1]\ J(t) \qquad (2)$$

where A is the ratio of the slope of the lines to the intercepts on the compliance axis. To satisfy equation 1, A must be independent of time.

The lines drawn in Fig. 2 were determined by the least squares method to fit the 12 points shown. The ratios of the slopes to the intercepts at various times are given in column 2 of Table I. The ratios increase slowly with time indicating that the data do not conform exactly to an expression of the form given by equation 2. There are systematic deviations from this simple expression; however, constant values for the ratio of the slope to the intercept are obtained from the straight lines fitted to the first six points (ie, over the range 10–60 N/m^2). These ratios are shown in column 3 of Table I.

Equation 2 expresses the nonlinear creep compliance as a perturbation from the limiting value as the strain approaches zero. The general form of such an equation is given by the Maclaurin series:

$$\mathcal{J}(\sigma,t) = \mathcal{J}(\sigma,t) + \sigma\left\{\frac{\partial \mathcal{J}}{\partial \sigma}\right\}_{\sigma=o} + \frac{\sigma^2}{2}\left\{\frac{\partial^2 \mathcal{J}}{\partial \sigma^2}\right\}_{\sigma=o} + \ldots\ldots \qquad (3)$$

Equation 2 is equivalent to the first two terms of this series if

$$A = \frac{1}{J(t)}\left\{\frac{\partial \mathcal{J}}{\partial \sigma}\right\}_{\sigma=o} \qquad (4)$$

It should be possible to fit the experimental data over a wider range of stresses by including additional terms. The results presented here, however, were not taken over a wide enough range of stresses nor with the necessary reproducibility to justify any attempt to fit an expression with higher order terms.

Analysis of the results for measurements on creep recovery lead to similar conclusions. The recovered strains, after various stresses had been applied for 250 sec, are plotted as a function of the recovery time in Fig. 3 using logarithmic scales. These curves are for the same six stresses for which the creep strains are shown in Fig. 1. Again the curves can be approximately superimposed by vertical shifts and the ratio of the recovered strain to the stress may, therefore, be represented by the product of a function of stress and a function of time similar to that proposed for the creep compliance in equation 1.

The plots of the strain divided by the applied stress for various recovery times are shown in Fig. 4. The straight lines shown were fitted to the 12 points by the least squares method. The ratios of the slopes to the intercepts for these lines are shown in column 3 of Table II. Except for the starting point, these ratios are constant for recovery up to a total time of about 350 sec but increase systematically for longer times. However, using the lines fitted to the first six points (10–60 N/m^2), the ratio, which is shown in column 4 of Table II, is equal to that found from the creep curves (column 3 of Table I). Thus it appears that the same stress function can be used to represent the dependence of both creep and recovery on the applied stress over a range of stress up to 60 N/m^2 and time up to 350 sec.

The strain remaining after creep and a long recovery time is a measure of the flow that has occurred during creep. For a sample with a yield value, this residual strain would be expected to be zero for stresses below the yield value and to increase with stress for stresses above the yield value. The plot of residual strain vs. stress (Fig. 5) is a continuous curve passing through the origin. There is no indication of a yield value that would correspond to an intercept on the stress axis. This is contrary to the conclusion drawn by Bloksma (1962) from an observation that at 14 N/m^2 (140 dynes/cm^2) there is virtually no residual strain, whereas at 17 N/m^2 (170 dynes/cm^2) there is a residual strain of about 0.26×10^{-3}. His curve for 17 N/m^2, however, has an unexplained irreproducible step so that this evidence for a yield value must be treated with caution. Bloksma also states that for stresses between 30 and 50 N/m^2, the reproducibility has an "uncertainty of about a factor of 1.5" and at lower stresses "the reproducibility is even poorer."

Bloksma (1962) implied that because the compliance curves are almost rectilinear at very low stresses, the response is predominately elastic. This interpretation, which has been repeated by Sherman (1970), is not valid. A rectilinear "creep" curve is characteristic of a purely viscous material. Pure elastic or delayed elastic behavior is characterized by an equilibrium compliance that is not evident in any of the published curves.

An expression of the form of equation 2, in which the effects of stress and time are separated, suggests that it may be possible to predict the dependence of the creep recovery behavior on the time for which the load is applied at a particular stress, provided that the nonlinear creep compliance at that stress is known over a longer time. Creep curves were measured for four samples over a period of 10,000 sec with an applied stress of 50 N/m^2. All the experimental results showed that the rate of strain was monotonically decreasing over the whole period. Although this is not obvious from the long-term creep curve on the scale used in this paper, it is evident from the creep curves drawn on a magnified scale and from detailed examination of the numerical results. The results reported by Smith and Tschoegl (1970) are presented on too small a scale and for too short a time period to justify their claim that steady-state

TABLE II
Ratio of Slope to Intercept of Straight Lines Fitted to Recovered Strain/Stress vs. Stress Plots

Total Time (sec)	Recovery Time (sec)	A_1 [a] (m^2/N)	A_2 [b] (m^2/N)
250	0.0	0.0129	0.0086
253.2	3.2	0.0157	0.0086
256.3	6.3	0.0154	0.0084
260	10	0.0153	0.0083
266	16	0.0154	0.0087
275	25	0.0154	0.0084
290	40	0.0154	0.0086
313	63	0.0158	0.0089
350	100	0.0158	0.0091
450	200	0.0171	0.0096
648	398	0.0193	0.0109
1,044	794	0.0229	0.0116
2,245	1,995	0.0327	0.0173
5,260	5,010	0.0443	0.0233

[a] A_1 for lines fitted 10–120 N/m^2.
[b] A_2 for lines fitted 10–60 N/m^2.

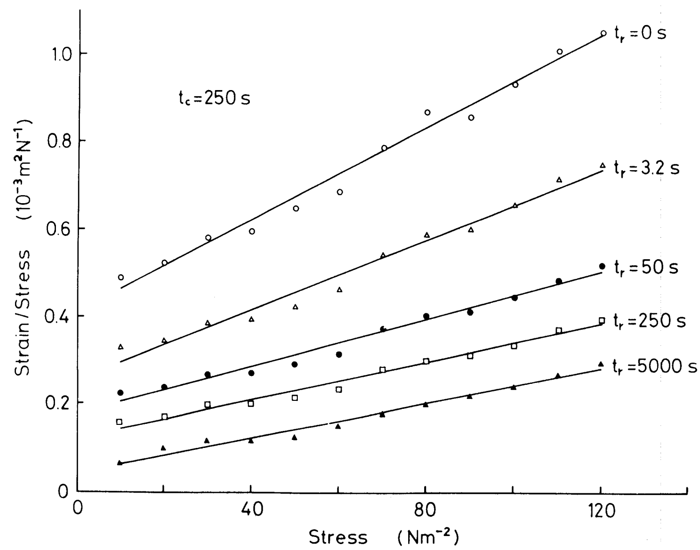

Fig. 4. Recovered strain divided by the applied stress plotted against the applied stress for various recovery times after load had been applied for 250 sec.

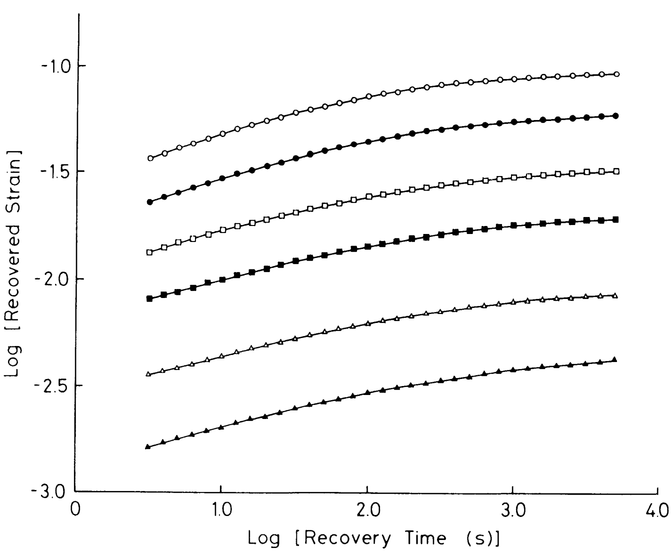

Fig. 3. Recovered strain plotted against recovery time for various applied stresses. ▲ = 10 N/m^2, △ = 20 N/m^2, ■ = 40 N/m^2, □ = 60 N/m^2, ● = 90 N/m^2, o = 120 N/m^2.

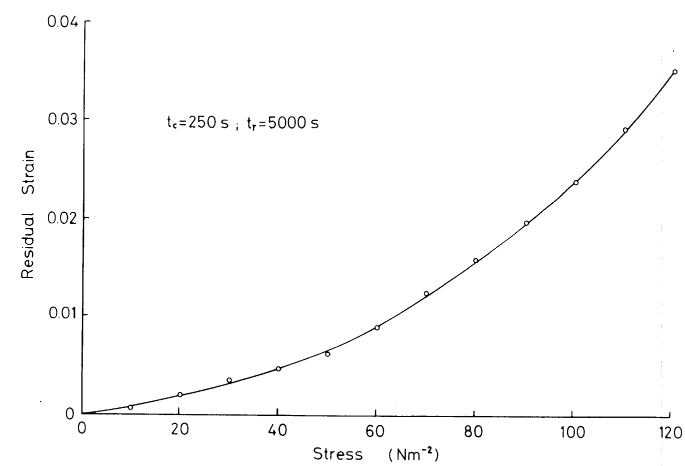

Fig. 5. Residual strain after creep for 250 sec and recover for 5,000 sec plotted against the applied stress.

flow has been achieved. Their plots do seem to have some curvature at long times even though they claim steady-state flow after about 1,500 to 1,800 sec.

A "master" creep function for a stress of 50 N/m^2 was obtained by the least squares method from the combined results of the four long-term creep experiments. This master creep function was used to predict the recovered strain after the stress was removed at various times assuming that the effects of loading and unloading by a single step of stress could be superimposed, ie,

$$\gamma(t)/\sigma = \mathcal{J}(\sigma,t) - \mathcal{J}(\sigma,t - t_c);\ t \geqslant t_c \qquad (5)$$

where γ is the strain, and t_c is the time for which the stress was applied. The recovered strain is given by

$$\gamma_r(t) = \sigma\ \mathcal{J}(\sigma,t_c) - \gamma(t);\ t \geqslant t_c \qquad (6)$$

The first term on the right-hand side of equation 6 is the creep strain at the time the load was removed.

Creep and creep recovery strains were measured for a series of creep times for a load of 50 N/m^2 and typical plots of the recovery are shown in Fig. 6 together with the master creep curve.

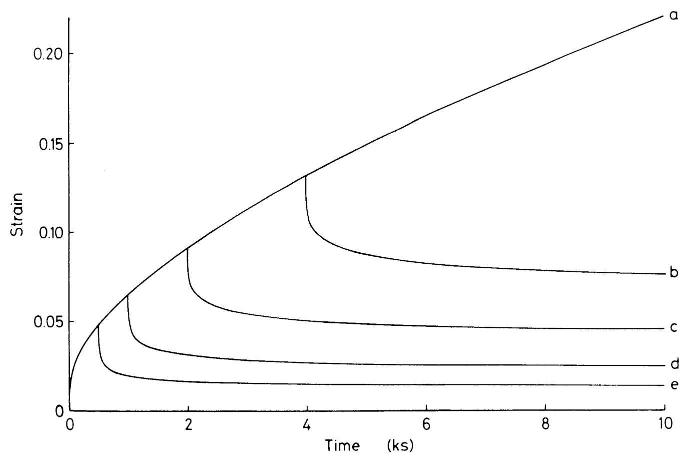

Fig. 6. Master creep curve (a) for an applied load of 50 N/m^2 together with recovery curves when load was removed after (b) 4,000 sec, (c) 2,000 sec, (d) 1,000 sec, and (e) 500 sec.

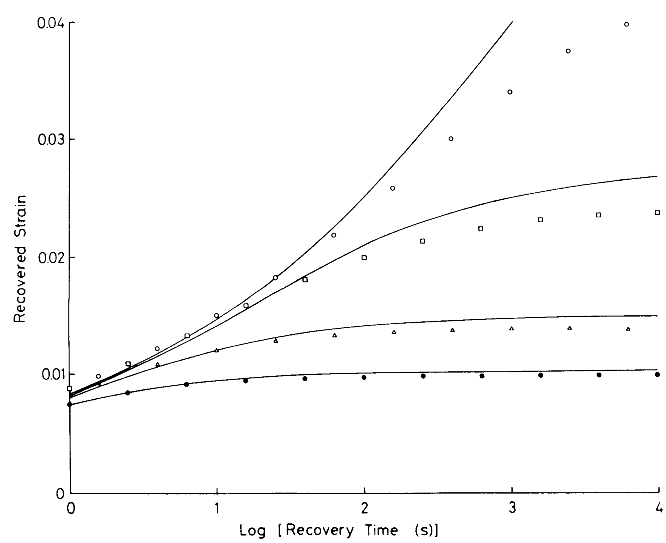

Fig. 7. Recovered strain when load of 50 N/m^2 was removed after various times plotted against recovery time. Continuous lines predicted using equation 6. Discrete points experimental values for creep times o = 2 sec, △ = 10 sec, □ = 100 sec, o = 1,000 sec.

Systematic differences of up to 6% from the master creep curve were observed for the individual creep curves. These are attributed to variations in mixing and loading the samples. The differences correspond to a small vertical shift in the log(strain) vs. log(time) plots. To correct for these small systematic errors, each individual creep and creep recovery curve has been multiplied by a factor, determined by the least squares method, to superimpose the creep part of the curve on the master curve.

The recovered strain after creep for different creep times predicted by equation 5 is shown by the continuous lines in Fig. 7 together with the experimentally determined points. It is clear that the fit is good when the total time of creep and recovery is short but the prediction fails for long recovery times, indicating that the assumptions used are valid only over a limited range of time.

The set of experiments, in which the applied stress was removed at various times and the strain remaining after a long recovery time measured, allows the creep strain at various times to be separated into recoverable (elastic or delayed elastic) strain and irrecoverable (flow or viscous) strain. These components of the creep strain are plotted in Fig. 8 for the series of experiments at 50 N/m^2. The irrecoverable strain is not directly proportional to the creep time indicating that the permanent deformation cannot be predicted by assuming a simple viscosity. At short times, the recoverable strain predominates; it requires creep for about 3,000 sec before the irrecoverable strain becomes greater than the recoverable strain at this particular stress.

It is not possible to divide the recoverable strain into instantaneous and delayed elastic components as, even at very short times, there is no discontinuity that would separate the instantaneous and delayed responses. The inertias of the sample and of the moving parts of the instrument prevent an instantaneous response. Consequently, the contributions from the instantaneous elastic response is predominant and claimed measurements of instantaneous elasticity from creep have depended on either instrumental artifact or measurements made at some arbitrary time after loading.

A series elastic element, to account for instantaneous elasticity, is common to many of the mechanical models proposed to approximate the behavior of dough. Models with a limited number of linear elastic and viscous elements, such as given by Bloksma (1972) and as implied by the "five fundamental parameters" of Yoneyama et al (1970), cannot explain the behavior of dough. More complex models, such as those presented by Muller (1975), have included frictional elements as well as additional linear elements in attempts to model the nonlinear behavior. The most complicated is that proposed by Glucklich and Shelef (1962b) with an infinite number of eight-element units each containing "three quite independent yield values." Tests of predictions based on these complex models have never been reported. It is most unlikely that such models could ever be useful.

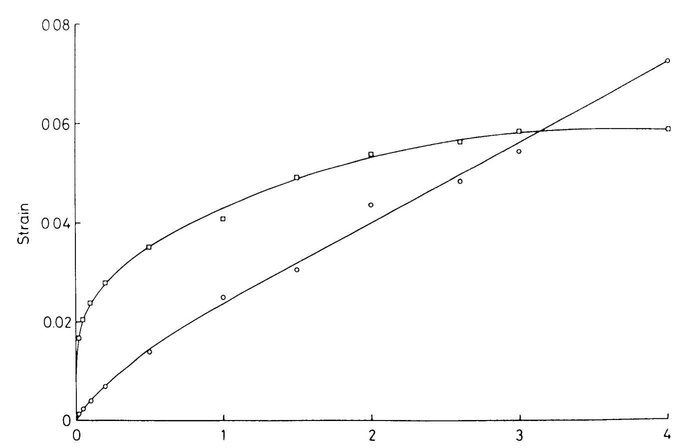

Fig. 8. Recovered creep strain (□) and nonrecovered strain (o) plotted against the logarithm of the creep time for t_r = 10 ks and σ = 50 N/m^2.

The results presented in this paper clearly show that it is possible to represent the mechanical response of a dough to a step in the deforming force over a limited range of stress and time by accepted rheological functions even though it is a nonlinear material. Further work is required to determine whether it is possible to obtain a useful characterization of the mechanical properties of dough by extending this approach to other rheological functions.

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