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ORIGINAL CONTRIBUTION

The effect of pre-test deformation on dough rheology

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Abstract We show that the strain involved in forming a dough specimen before testing will often radically alter the measured rheological properties in shear and in elongation if these pre-strains are greater than about 0.5 (Hencky strain). It is shown that this may be accounted for by changing the G(1) value used in the damage function model to a relevant value.

Keywords Dough rheology · Pre-test deformation · Shearing · Uniaxial elongation · Storage modulus · Damage function · Lodge model

Introduction

Typically, a dough sample, after mixing, is rested for 45 min; then, it is shaped to form specimens for testing and then rested again so that any initial stresses may dissipate. The actual rheological testing then commences; the softening ("kneading") due to deformation during testing occurs, and theoretical work whereby a "damage function" is introduced (Tanner et al. 2007, 2008a, b, 2011) permits a rheological description which is reasonably accurate for predicting the stresses in the specimens. However, it has become clear that there is also a dependence of the measured rheological properties of the dough due to the deformation involved in forming the specimens after mixing and before testing. Davidou et al. (2008) noticed this in their cone/plate and parallel-plate tests, but they did not tie the observed increases in $|G^*|$ to the amount of the pre-test deformation in a quantitative manner. Dai and Tanner (2012) suggested that the increased resistance to deformation in small-diameter specimens in elongation was due to pre-stretching during the manufacture of the specimens. The smaller 5-mm-diameter specimens were about twice as resistant to deformation as the larger ones of 20-mm diameter, and a rough relation linking the pre-test strain to the measured properties was proposed.

In this paper, we present some new experiments and we seek to describe this pre-deformation hardening phenomenon more precisely, not only in elongation but also in shear deformation.

Materials and methods

Dough preparation

A commercial Australian flour was used in this study. The flour sample was variety Janz wheat, grown in 2001 at Narrabri, NSW and milled on a Buhler experimental mill. It is a benchmark Australian hard kernel wheat, with medium dough strength. The dough was produced in a 10-g mixograph by mixing 200 mg of salt, 6.0 g of distilled water and 9.5 g of flour. The sample was mixed to a pre-determined mixing time (peak dough development) of 3 min, by four planetary pins on the head revolving round three stationary pins on the bottom of the mixing bowl. The rotation speed was measured to be 71 rpm. The mixing operation was conducted at a temperature of 24 °C and under ambient humidity in an air-conditioned laboratory. The dough used for the oscillatory and steady shear measurements was

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stored in a sealed bag after mixing and was allowed to relax for 45 min.

In the case of oscillatory measurements without pre-strain, the mixed dough was immediately placed on a Teflon plate in a form of a half cylinder. The Teflon plate was then placed in an airtight container and placed in a freezer for 1.5 h at −20 °C.

Small-strain shear frequency sweep tests

We begin with small-strain (0.1 %) oscillatory tests. The frequency sweep experiments were carried out on a Paar Physica MCR 301 rheometer. These tests (and the steady shear tests discussed below) were carried out at 24 ± 1 °C with and without pre-strain of the sample. Reported results are the median of (usually) five tests.

In the first set of experiments, dough was mixed to peak dough development and was stored in a sealed bag after mixing, and it was allowed to relax for 45 min. Parallel plates with a diameter of 25 mm were used, and the gap was set to 1, 2 and 5 mm for the measurements. Slippage during testing was prevented by two pieces of sandpaper that had been glued to the parallel plates. The sample was mounted on the lower plate (the average height of the sample after mounting was about 29 mm) and compressed between the plates by moving down the upper plate to set gaps of 1, 2 and 5 mm, respectively. Excess dough was trimmed, and the edge of the sample was coated with Shell® petroleum jelly to prevent moisture loss. After that, the sample was allowed to relax for a further period of 45 min. For oscillatory shear measurements, the frequency sweeps in the frequency range of 0.01–30 Hz were conducted in the linear response range at a strain amplitude of 0.1 %. After testing, the sample was unloaded and a new sample was loaded for the next frequency sweep.

In the second set of experiments, where the pre-strain on the sample was to be minimised, the dough placed in the freezer was taken out after 1.5 h and cut to a disc approximately 2.1 mm in thickness with a device in which a sharp blade was mounted. This disc was further rested at ambient temperature for 45 min. The parallel plates with a diameter of 25 mm were used, and the gap was set to 2 mm for the measurements. Slippage during testing was prevented by two pieces of sandpaper as described above. After 45 min, the disc of dough was mounted on the lower plate and compressed between the plates by moving down the upper plate to a set gap of 2 mm. Excess dough was trimmed, and the edge of the sample was coated with Shell® petroleum jelly to prevent moisture loss. The sample was further rested for 45 min and frequency sweeps in the frequency range of 0.01–30 Hz were conducted at the strain amplitude of 0.1 %. After testing, the sample was unloaded and a new sample was loaded for the next frequency sweep.

Steady shearing

The steady shearing tests were carried out in a similar way on the rheometer; the gap was set at 1, 2 and 5 mm and sandpaper was used to avoid slip. A constant rim shear rate of 0.1 s^{-1} was applied, and the shear stress was reported as a function of strain. No tests were carried out with the very small pre-strain; at least two tests were made for each gap setting.

Results of shear testing

The results of the small-strain tests with various gap sizes are shown in Fig. 1, where the storage modulus G' is plotted as a function of frequency (in radians per second). The degree of pre-test strain can be calculated; from an initial height of about 29 mm (h_0) to the final gap (h), the negative Hencky pre-strain is compressive in the axial direction and is given by $\ln(h/h_0)$. However, in order to correlate with our previous tensile experiments (Dai and Tanner 2012), we will use the maximum positive Hencky strain (radial direction) of ε_0 which is defined as

$$\varepsilon_0 = \frac{1}{2} \ln \left(\frac{h_0}{h} \right). \tag{1}$$

The value of ε_0 for the special sample, which is nominally without pre-strain, is about 0.02, which is believed to be negligible. The "stiffening" of the samples is clear from Fig. 1, as judged by the variation of G' at any given frequency with variable pre-strain. There is roughly a 180 % increase in G' when pre-strain rises from essentially 0 to 1.7. The results for G'' are similar. These effects are comparable in magnitude to those found by Davidou et al. (2008).

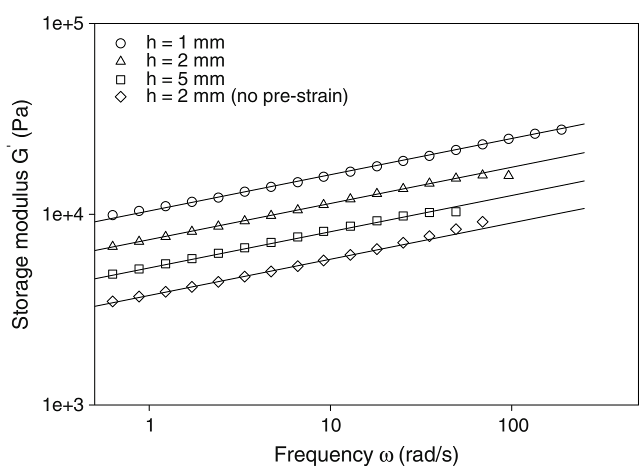

Fig. 1 Showing the increase of the storage modulus G' with increased pre-strain. The *bottom curve* has essentially no pre-strain (∼0.02); and in ascending order, the positive pre-strains in the radial direction (Hencky strains) are estimated to be 0.88, 1.34 and 1.68

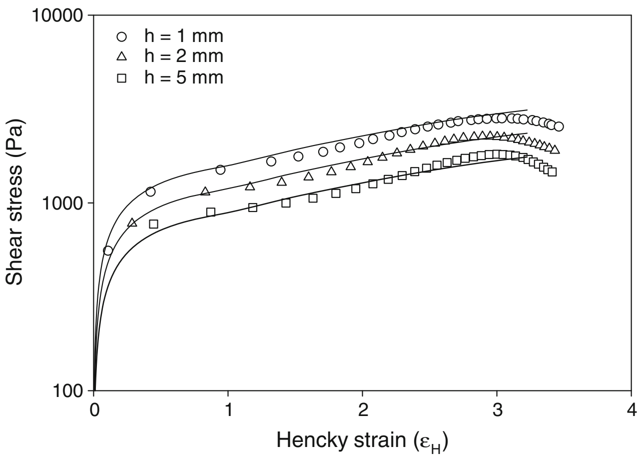

Fig. 2 Showing the shear stress as a function of pre-strain with pre-strains of 0.88, 1.34 and 1.68 as in Fig. 1; the constant shear rate was 0.1 s^{-1}

For the steady shearing (Fig. 2), a similar stiffening occurs—the magnitudes are very similar to the G' and G'' increases.

Hence, there is no doubt that the history of strain during specimen preparation has a profound effect on the dough rheology, both in shear and in elongation.

An experimental correlation

We can display all the results in Figs. 1 and 2 and our previous tensile tests as the ratio R of the measured properties as a function of the specimen pre-strain ε_0. For the small-strain oscillatory measurements, we use

$$R = \frac{G'(\omega, \varepsilon_0)}{G'(\omega, 0)}, \tag{2}$$

while for the shearing tests, we use

$$R = \frac{\tau(\varepsilon_H, \varepsilon_0)}{\tau(\varepsilon_H, 0)} \tag{3}$$

where the zero pre-strain result has been estimated from Fig. 1.

For the tensile tests done previously (Dai and Tanner 2012), we use, from Fig. 3,

$$R = \frac{\sigma(\varepsilon_H, \varepsilon_0)}{\sigma(\varepsilon_H, 0)} \tag{4}$$

with $\varepsilon_H = 2.0$, where σ is the tensile stress. All of these results for R are plotted in Fig. 5. We see that for pre-strains of about 0.5 or less, there is not much change in rheology, as what we have concluded previously (Dai and Tanner 2012). For larger pre-strains, considerable changes occur.

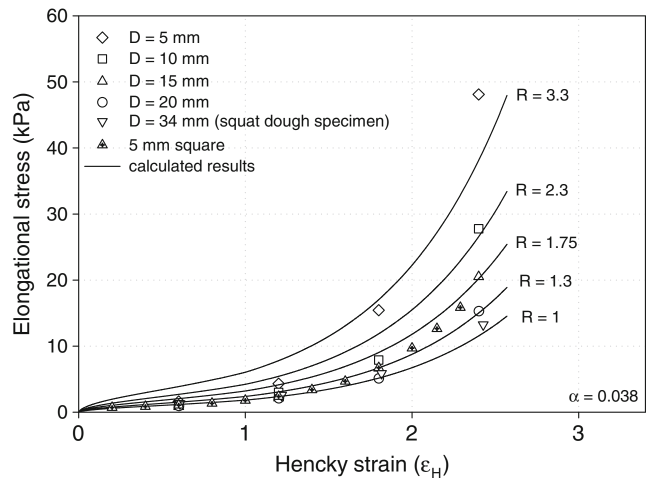

Fig. 3 The elongational tests: the 5 mm square specimen has essentially zero pre-strain; the other specimens have the following estimated pre-strains: 5-mm diameter, 2.46; 10 mm, 1.22; 15 mm, 0.87; and 20 mm, 0.76. The 34-mm-diameter specimen is estimated to be at less than 0.3 pre-strain (Dai and Tanner 2012). The ratio R is shown on the fitted curves. The rate of extension was 0.01 s^{-1} in all cases

Theoretical implications

We will consider the implications of these results for the damage function model (Tanner et al. 2007, 2008a, b, 2011) which has been used to describe the rheological behaviour of bread dough. For convenience, we briefly review the proposed model, which is based on the Lodge model (Lodge 1964) with the addition of a damage function and it is given by

$$\boldsymbol{\sigma} = -P\mathbf{I} + f \int_{-\infty}^{t} m\left(t - t'\right) \mathbf{C}^{-1}(t')dt', \tag{5}$$

where $\boldsymbol{\sigma}$ is the total stress tensor, m is a memory function, P is pressure, $\mathbf{I}$ is a unit tensor, $\mathbf{C}^{-1}(t')$ is the Finger strain tensor (Tanner 2000) at time t', calculated relative to the configuration at the present time t and f is a damage function, which is a function only of the deformation (Hencky strain ε_H). The damage function for the Janz variety dough used here is shown in Fig. 4. The range of the damage function is $0 < f \leq 1$, ranging from no damage ($f = 1$) to failure of the specimen ($f = 0$). Clearly, if the damage function $f = 1$, then Eq. 5 is Lodge's rubber-like model (Lodge 1964).

Following Winter and Mours (1997) work on polymer gels and the work of Gabriele et al. (2001) on foods, the memory function m for dough is typically assumed to be of a power-law form and it can be expressed as

$$m(t) = pG(1)t^{-(p+1)}, \tag{6}$$

see also Tanner et al. (2011) for other applications and references to this memory form.

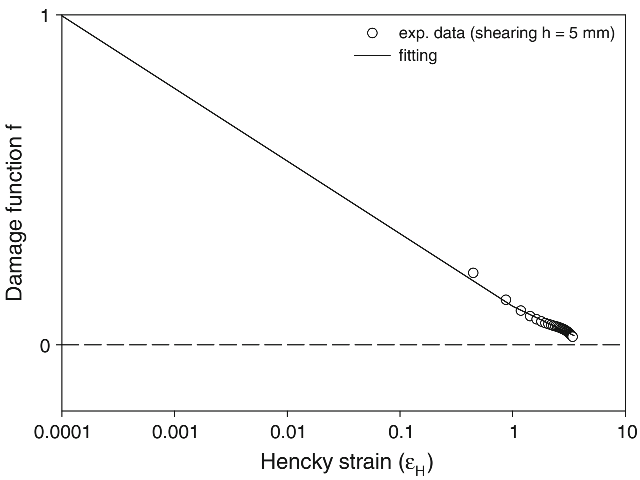

Fig. 4 A typical damage function for Janz dough as a function of strain; at a small strain of 10^{-4}, there is no appreciable damage and $f = 1$

In Eq. 6, p is a power-law exponent depending on the dough type; the value of p is usually in the range 0.18–0.3 (Morgenstern et al. 1996; Phan-Thien et al. 2000; Tanner et al. 2007, 2008a) and $G(1)$ is a constant with the dimension Pa-s^p. In the linear region, at very small strains (say $\sim 10^{-3}$), the shear relaxation modulus $G(t)$ corresponding to the power-law memory function of Eq. 6 is known to be of the form (Pipkin 1986)

$$G(t) = G(1)t^{-p}. \tag{7}$$

Note that the constant $G(1)$ in Eq. 7 is numerically equal to the value of the shear relaxation modulus at $t = 1$ s, but it has different dimensions from $G(t)$; also, we have the memory function $m = -dG/dt$.

To describe a wider range of deformation patterns, the constitutive model mentioned above has been developed and discussed in Tanner et al. (2011), especially for biaxial extension (spherical bubble test). In the improved model, a Mooney–Rivlin term (with the Cauchy–Green strain tensor **C**) is included. The improved model is given by

$$\boldsymbol{\sigma} = -P\mathbf{I} + \frac{f}{1+\alpha} \int_{-\infty}^{t} m\left(t - t'\right)\left[\mathbf{C}^{-1}(t') - \alpha \mathbf{C}\left(t'\right)\right] dt'. \tag{8}$$

Here, α is a constant (here taken to be 0.038, Tanner et al. 2011) and $\mathbf{C}(t')$ is the Cauchy–Green strain tensor at the previous time t'. For elongational flows of incompressible materials with a constant deformation rate, for example, uniaxial elongation and biaxial extension, the Finger strain tensor has the form

$$\mathbf{C}^{-1}(t) = \begin{bmatrix} e^{2\dot{\varepsilon}t} & 0 & 0 \\ 0 & e^{-(2-q)\dot{\varepsilon}t} & 0 \\ 0 & 0 & e^{-q\dot{\varepsilon}t} \end{bmatrix} \tag{9}$$

and the Cauchy–Green tensor is

$$\mathbf{C}(t) = \begin{bmatrix} e^{-2\dot{\varepsilon}t} & 0 & 0 \\ 0 & e^{(2-q)\dot{\varepsilon}t} & 0 \\ 0 & 0 & e^{q\dot{\varepsilon}t} \end{bmatrix}, \tag{10}$$

where $\dot{\varepsilon}$ is a constant strain rate and q is a constant; $q = 1$ for steady simple elongation and $q = 4$ for biaxial extension (Tanner 2000).

The damage function can be found from a steady shear test with a constant shear rate beginning from rest (5-mm gap shearing test). At very small deformations, $\varepsilon_H \leq 10^{-4}$, the damage function $f = 1$. Then, the damage function is of a logarithmic form (here, we use logarithms to base 10) in terms of the Hencky strain, which is presented as follows:

$$\begin{aligned} f &= -a \log \varepsilon_H + c \quad \varepsilon_H \leq 1 \\ f &= -b \log \varepsilon_H + c \quad \varepsilon_H > 1 \end{aligned}. \tag{11}$$

where a, b and c are positive constants; a typical example of f for Janz dough is shown in Fig. 4, in which $a = 0.22$, $b = 0.163$ and $c = 0.117$.

In the case of shear flow, the relationship between the shear strain γ and the Hencky strain ε_H is given by

$$\varepsilon_H = \ln\left[\frac{\gamma}{2} + \sqrt{1 + \frac{\gamma^2}{4}}\right]. \tag{12}$$

As discussed in Tanner et al. (2011), the improved model Eq. 8 was used to describe a wide range of deformation patterns, which include small-strain deformations, relaxation of stress after a single suddenly applied step of shear, simple shearing beginning from rest, biaxial stretching beginning from rest, constant-rate elongation beginning from rest, sequences of step strains, large-amplitude sinusoidal strains and recoil after elongation. The model was also used to describe creep flow by Dai et al. (2011).

From the experiments reported above, we see the effect of pre-strain on rheology; the same behaviour was found in elongational tests using a thin sheeting noodle specimen and also with the specimen shape used in the well-known Kieffer test (Tanner et al. 2009; Dunnewind et al. 2004). In the Kieffer test, the cross-sectional area of specimens is about 20 mm^2 which is in the same order as the 5-mm-diameter specimens. As discussed above, the influence on the stress–strain behaviour is actually not the size of specimens, but the pre-deformation which takes place during the making of the specimens. The processing for making these small-size dough specimens is quite complicated, for example, in the biaxial extension (spherical bubble) test. For a complex material like dough, the pre-deformation has to be taken into account and cannot be neglected if the pre-deformation is larger than a critical value. The critical value of the pre-deformation suggested from elongation tests is about 0.5 Hencky strain units (Dai and Tanner 2012). Since the pre-deformation is not taken into account in the model Eq. 8, the model therefore fails to describe the effect of pre-deformation on bread dough rheology. We seek here to understand and describe these effects—evidently, from Eq. 8 either f, m or the strain measure could be affected by the pre-test deformation, and we shall consider all of these possibilities. For the method of calculation of the results, the details are given by Tanner et al. (2011).

Pre-strain in the constitutive model

In the model Eq. 8, the Finger strain tensor $\mathbf{C}^{-1}(t')$ and the Cauchy–Green strain tensor $\mathbf{C}(t')$ are the relative strain measures at current time t, calculated relative to the previous configuration at time t'; the flow starts at time $t = 0$. It is clear that the pre-deformation in making specimens is not included in these strains. One idea to be tried is that the pre-strain is simply inserted into the Finger strain tensors $\mathbf{C}^{-1}(t')$ and Cauchy–Green strain tensor $\mathbf{C}(t')$. The model with pre-strain is therefore given by

$$\boldsymbol{\sigma} + P\mathbf{I} = \frac{f}{1+\alpha}\int_{-\infty}^{t} m(t-t')\mathbf{S}(t')dt', \tag{13}$$

where $\mathbf{S}(t')$ is a strain tensor and is defined as

$$\mathbf{S} = \mathbf{F}^{-1}\mathbf{C}_0^{-1}\mathbf{F}^{-T} - \alpha\mathbf{F}^T\mathbf{C}_0\mathbf{F}, \tag{14}$$

in which $\mathbf{C}_0^{-1}$ and $\mathbf{C}_0$ are the Finger strain tensor and the Cauchy–Green strain tensor of the pre-deformation, respectively; $\mathbf{F}$ is the deformation gradient tensor. To define $\mathbf{F}$, let $\mathbf{x}(t)$ be the current position of a particle at time t and $\mathbf{r}(t')$ is the position of the particle at an earlier time t'. Then, the deformation gradient tensor can written as $\mathbf{F} = \partial\mathbf{r}/\partial\mathbf{x}$ or $F_{ij} = \partial r_i/\partial x_j$; the Finger strain tensor and the Cauchy–Green strain tensor are defined by $\mathbf{C}^{-1} = \mathbf{F}^{-1}\mathbf{F}^{-T}$ and $\mathbf{C} = \mathbf{F}^T\mathbf{F}$, respectively (Tanner 2000). For the pre-deformation produced by a non-shearing flow, the Finger strain tensor can be written as

$$\mathbf{C}_0^{-1} = \mathrm{diag}\left(e^{2\varepsilon_0}, e^{-(2-q)\varepsilon_0}, e^{-q\varepsilon_0}\right), \tag{15}$$

and the Cauchy–Green strain tensor is

$$\mathbf{C}_0 = \mathrm{diag}\left(e^{-2\varepsilon_0}, e^{(2-q)\varepsilon_0}, e^{q\varepsilon_0}\right). \tag{16}$$

where ε_0 is the pre-strain (Hencky strain).

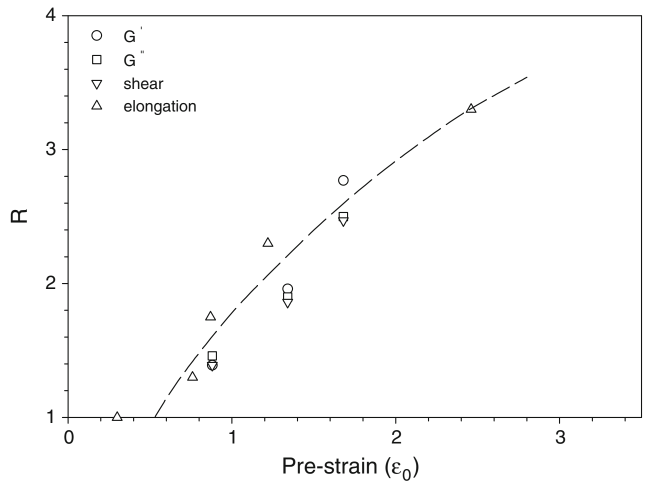

Fig. 5 The stiffening parameter R as a function of pre-strain for all tests. For a pre-strain of less than 0.5, the value of R is close to unity. The suggested correlation for Janz dough is indicated by the *dashed line*

While the idea of including a pre-strain factor $\mathbf{C}_0$ as in Eq. 14 works well in predicting the extra resistance in small-diameter tensile tests (including our Kieffer test experiments (Dunnewind et al. 2004; Tanner et al. 2009)) and biaxial extensions (Tanner et al. 2011), it fails to predict the strengthening in shearing; in fact, the modified strain shows that shear resistance must decrease with increasing compressive pre-strain. Let us suppose that a piece of dough is compressed between parallel plates before testing. The experimental results display the measured increase of the storage modulus G' (Fig. 1.), yet the pre-strain theory discussed above in Eq. 14 shows a decrease must occur in shear resistance. Hence, this theory is inapplicable.

From Fig. 2, we deduce that the damage function f in shearing is not substantially changed by pre-straining, since all curves have the same shape. Hence, we are forced to conclude that the main effect of pre-strain is to alter the memory function m (Eqs. 6 and 7).

Now, the small-strain response is (Pipkin 1986)

$$G' = G'(1)\omega^p \tag{17}$$

where $G'(1)$ is a multiple of $G(1)$. From Fig. 1, the slope p of the curves is not affected by the pre-strain; hence, it suffices to write $G(1, \varepsilon_0)$ instead of $G(1)$ in Eqs. 6 and 7. The approximate variation of $G(1, \varepsilon_0)$ with pre-strain is given as the dashed curve in Fig. 5; since G' is a multiple of $G(1)$, it too is affected by pre-strain.

Conclusions

Provided the relevant $G(1,\ \varepsilon_0)$ value is used, the damage function model does not need to be changed and may be used for predicting dough behaviour. Pre-strains greater than about 0.5 (Hencky measure) will make large changes in $G(1)$ due to the structural changes. One is reminded of the early work of Schofield and Scott Blair (1932) who showed that dough recoiled (almost) completely for strains of less than 30 %. We believe that the increased resistance to deformation in all cases is due to alignment of the oblate spheroidal starch particles along the positive strain direction, which acts to "reinforce" the material for strains of the order of 0.5 and greater. Such a mechanism can also be expected in other soft solids with non-spherical embedded particles.

We found previously (Tanner et al. 2009) that the well-known Kieffer test results (Dunnewind et al. 2004) were not consistent with the $G(1)$ values measured from larger diameter tensile specimens—the Kieffer results were larger. Because the Kieffer method of sample preparation is similar to that with our 5-mm-diameter specimens, we believe that the stress enhancement in this test can be explained by the pre-strain history. Similarly, a flat specimen of thin rectangular initial cross section showed increased resistance due to the work done during preparation. The bubble test (Tanner et al. 2011) also requires rolling to produce the initial specimen shape and shows a consequent increased resistance to deformation.

Specimen preparation usually requires compression in one direction and elongation in others. This may operate on the structure of the dough in at least two ways. One, it may operate to align the non-spherical A starch particles hence reinforcing the dough strength (Dai and Tanner 2012) or it may consolidate the dough by closing up defects due to the pressure applied during specimen shaping. The exact mechanism remains to be discovered.

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