# Particle-Size Related Physical Properties of Flour Produced by Smooth Roll Reduction of Hard Red Spring Wheat Farina<sup>1</sup>

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#### **ABSTRACT**

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Hard red spring wheat farina was reduced at all possible combinations of three roll gaps and three differentials using a roll stand of the 254-mm Grain Research Laboratory experimental mill equipped with smooth frosted rolls. The flour produced was separated into three particle-size fractions by sieving through 91- and 53- $\mu$ m aperture sieves. The <53- $\mu$ m fraction exhibited much greater starch damage than the coarser fractions. Increasing differential and decreasing roll gap increased starch damage for chop and the two coarser fractions. Greater compressive stresses (from reduced roll gap) did not affect the starch damage of the <53- $\mu$ m fraction, possibly because greater stress caused more fracturing rather than altering the mode of particle release from the parent farina. The fracture mode is thought to start at the starch granule-protein interface. Increased

differential increased the starch damage in the <53- $\mu$ m fraction. Shear forces applied by the differential at the starch-protein interface would alter the mode of fracture. The ratio of starch damage estimated by amylose extractability to starch damage estimated by  $\alpha$ -amylase digestibility increased with increasing differential for all three particle-size fractions, substantiating the hypothesis that differential would initiate crystallite slippage in the amorphous regions of the granule. Higher flour ash and darker flour color values at 1:1 differential were explained as a consequence of brittle fracture occurring through the more proteinaceous regions, since at 1:1 differential crystallite movement is inhibited due to reduced shearing forces.

Hard red spring wheat farina is composed primarily of endosperm, but also contains adhering bran and germ. The main components of the endosperm cells are starch and protein. Starch granules are mainly semicrystalline polymeric entities of branched amylopectin and amorphous linear phases of amylose (Lineback 1984). The protein is a heterogeneous mixture of components varying in molecular weight and amino acid composition (Bushuk and Wrigley 1974). Within a given endosperm cell, a complex interface of interdigitating starch and protein forms due to the outward growth of the starch granules compressing the protein bodies as the kernel develops (Simmonds and O'Brien 1981).

Farina particles passing through the grinding zone of the roller mill are subjected to applied shear forces from contact between points on the particles and the roll surfaces, and compressive forces on the particles as a whole. Compression alone induces a complex stress distribution of shear and normal stresses (Hooper 1971). Differential superimposes a secondary force perpendicular to the applied compression (Kozmin 1921), further complicating the stress distribution in the particles. The reduction in size resulting from farina particle fracture occurs after a given amount of deformation. This deformation (or strain) will be ductile or brittle depending on the applied stresses and the farina components upon which the stresses act.

The number, types, and properties of flour particles formed are dependent on the manner of fracture and where it occurs. For example, mechanical starch damage is only manifest after

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disruption of the crystalline structure of the starch granule (LeLievre 1974).

A previous paper (Scanlon and Dexter 1986) related the effects of roll speed, feed rate, and differential to the properties, especially starch damage, of flour produced by smooth roll reduction of hard red spring wheat farina. Differential exerted the greatest effect on starch damage, determined by susceptibility to  $\alpha$ -amylase digestion (Farrand 1964) and by extractability of amylose (McDermott 1980). This was surmised to result from shear forces acting on the particle surface causing fracture by shear yielding through the starch granules. The resulting small particles would suffer crystalline deformation during shear yielding and would contribute disproportionately to the starch damage value of the flour as a whole. Greater differential would increase the proportion of these particles, thus increasing the flour starch damage. An increase in the ratio of amylose extractability to amylase digestibility was observed as differential increased and particle size decreased (Evers et al 1984); this was thought to be due to the large surface area/volume ratios of the fine particles allowing greater penetration of water and increased leaching of amylose.

This study attempts to identify the effects on flour properties of different applied forces (i.e., compressive and shear) from the effects of flour particle size. Hard red spring wheat farina was reduced under various combinations of roll gaps and roll differentials to alter the magnitude and relative contributions of compressive and shear forces. The flour samples obtained were fractionated into three particle size ranges, and compositional changes and properties were quantified for each flour fraction and the composite flours.

#### MATERIALS AND METHODS

## Farina Preparation

Farina was prepared as previously described (Scanlon and Dexter 1986). Using a Buhler MLU 300 laboratory plansifter, it was determined that 85% of the farina was in the 132-247  $\mu$ m size

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range. Moisture content, protein content, ash, and Kent-Jones color grade were 14.6%, 12.6%, 0.45% (14% moisture basis), and 1.1, respectively.

## **Milling Conditions**

Milling was performed with the reduction roll stand of the Grain Research Laboratory (GRL) Ross Research mill (Black et al 1980). The rolls have a frosted finish and are 254 mm (10 in.) in diameter with an effective grinding length of 115 mm (4.5 in.).

Differentials of 1.0:1, 1.5:1, and 2.0:1 were used at a constant slow roll speed of 390 rpm. The two roll gaps to be used in addition to the 38- $\mu$ m (0.0015-in.) roll gap used previously (Scanlon and Dexter 1986) were selected on the basis of the work required to implement size reduction.

The roll gap was set at 38  $\mu$ m and the rolls were heated to 40° C, the stable operating temperature. The feed rate used throughout was 4.0 kg/m per minute. This was the maximum feed rate that did not cause knocking as stock was ground. This knocking, indicative of overloading of the grinding zone, creates uncertainty in the size of the roll gap and was most acute at a differential of 1:1. The power consumed as stock was ground at a roll gap of 38  $\mu$ m and a differential of 1.5:1 was recorded, and the roll gap was progressively decreased until the power level during grinding increased by 50%. After the rolls cooled to room temperature (21° C), this small roll gap was found to be 30  $\mu$ m (0.0012 in.). At a differential of 1.5:1, the roll gap was increased until the power level during grinding decreased by 50% compared to the power consumed at the initial roll gap (38  $\mu$ m). At 21° C the large roll gap was 63  $\mu$ m (0.0025 in).

## **Grinding Experiments**

The rolls were preheated to  $40^{\circ}$  C. Millings were performed on lots of 500 g of farina for each differential and replicated four times, not in a random manner, but in a fixed pattern to counteract the heating of the rolls that occurred if high-differential millings were performed successively. For the small and intermediate roll gap (30 and 38  $\mu$ m at 21° C), an extra lot of 500 g of farina was milled for each replicate of the 1:1 differential series to compensate for the low flour production. After sifting (see below), the two duplicate flours from each replicate were combined and mixed.

For the large roll gap experiment  $(63 \mu m)$ , low flour production was obtained at all differentials. In this case all three differentials were replicated four times in random manner, and these 12 millings were repeated the next day. The flours obtained from identical replicates on different days were combined and mixed.

Milling energy consumption during all grinding runs was determined from a watt transducer connected to the drive motor of the roll stand (Kilborn et al 1982).

## Flour and Flour Fractions Preparation and Characterization

Milled stock was blended and sifted for 2 min on an Allis-Chalmers box sifter over a 10XX ( $136\,\mu\text{m}$ ) bolting cloth to obtain the composite flour for analysis. Any duplicate flours were combined, and  $100\,g$  of each flour was removed for sifting into the three fractions. Sifting was performed on a Buhler MLU 300 laboratory plansifter for 4 min with sieve openings of 91  $\mu\text{m}$  (15XX) and 53  $\mu\text{m}$  (53 Nitex). The largest starch granules have diameters up to  $40\,\mu\text{m}$  (Hoseney 1986), and although mechanically damaged granules are less even in outline (Williams 1970), the 53- $\mu\text{m}$  Nitex sieve should separate most free starch granules. Stock held on each sieve and the pan was weighed and designated as coarse fraction ( $91-136\,\mu\text{m}$ ), medium fraction ( $53-91\,\mu\text{m}$ ), and fine fraction ( $53-91\,\mu\text{m}$ ).

Starch damage for each flour and the three fractions was estimated by the enzymic method of Farrand (1964) and the nonenzymic method of McDermott (1980). For the Farrand method, sample size was reduced from 5 to 1 g for the fine fraction to obviate the longer digestion time required for samples of high starch damage (Williams and LeSeelleur 1970). Water absorption capacity was determined by the ultracentrifuge method of Preston and Tipples (1978). Protein determination of all samples was by the Kjeldahl method as modified by Williams (1973). Differential

scanning calorimetry (DSC) was performed on composite flours and the fine fraction, using a DuPont 9900 Thermal Analyser equipped with a DuPont 910 cell base and a pressure calorimeter cell according to the method of Biliaderis et al (1985). Analyses were performed in triplicate using sample weights of 8–12 mg (20% solids; aqueous flour suspensions). Apparent gelatinization enthalpies ( $\Delta H$ ; J/g) were determined using the DuPont software analysis programs.

The composite flour samples were analyzed for color with a Kent-Jones and Martin series 2 flour color grader (Holas and Tipples 1978) and for ash by AACC method 08-01 (1983).

All analytical data were corrected to 14% moisture basis.

#### Statistical Analysis

Data were analyzed by analysis of variance (ANOVA) using factorial design (Snedecor and Cochran 1967). The significance of differences between treatments was tested by the method of Duncan (1955).

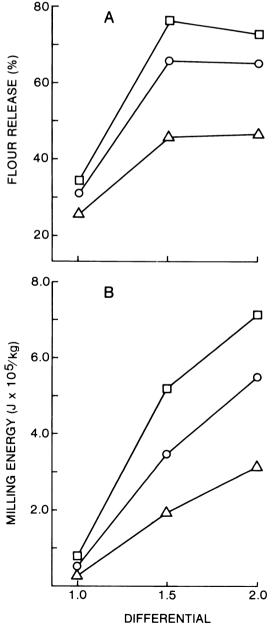


Fig. 1. Effect of roll differential at roll gaps of  $63 \,\mu$ m ( $\Delta$ ),  $38 \,\mu$ m (O), and  $30 \,\mu$ m ( $\square$ ) on A, flour release and B, energy consumed during reduction of hard red spring wheat farina.

#### RESULTS AND DISCUSSION

## Flour Components and Their Properties

Flour is a heterogeneous collection of particle sizes. Examination of the composition and properties of flour as a whole will not allow detailed consideration of the manner in which fractures passed through components in the farina during size reduction. However, the changes induced in the flour as roll gap and differential are altered will reflect the effect of shear and compressive forces acting on the farina particles as they pass through the grinding zone.

Figure 1A shows the highly significant effects (P < 0.001) of roll

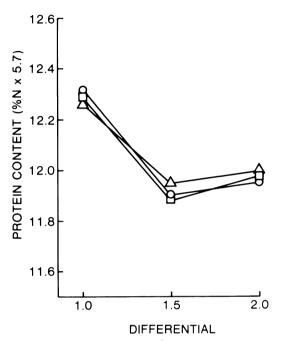


Fig. 2. Effect of roll differential at roll gaps of 63  $\mu$ m ( $\Delta$ ), 38  $\mu$ m (O), and 30  $\mu$ m ( $\square$ ) on the protein content of flour produced during reduction of hard red spring wheat farina.

gap and differential on the proportion of flour released. As roll gap decreases greater stresses are imposed, increasing the number of fractures, thus creating more flour particles at all differentials.

At 1:1 differential the only shear forces acting will be those arising as components of the applied compressive forces. The effect of the increased shear forces, imparted by increasing the differential from 1:1 to 1.5:1, was to increase flour yield. A similar result was seen by Evers et al (1984) as differential was increased from 1:1 to 1.25:1. Further increasing the magnitude of the shear forces imparted by the differential had little effect on flour release. In fact, at the small roll gap increasing the differential to 2:1 decreased the amount of flour formed.

In contrast, differential is more linearly related to the energy consumed during farina reduction (Fig. 1B). Energy consumption is significantly (P < 0.001) related to both roll gap and differential.

Protein content of the flour (Fig. 2) was significantly affected by differential (P < 0.001). Roll gap did not exert a significant effect (P > 0.05). The relationship between flour protein content and roll differential was similar to the relationship between flour release and roll differential; a large change from 1:1 to 1.5:1 and little change when differential was increased further.

Figure 3 shows the stress induced changes in flour properties. Enzymatic (Fig. 3A) and nonenzymatic starch damage (Fig. 3B) assays showed highly significant (P < 0.001) relationships of roll gap and differential to starch damage. Increased compressive stress loading of the farina caused more starch damage as did increased applied shear forces. These trends are consistent with the increased energy required to reduce farina as roll gap decreased and differential increased (Fig. 1B). Water absorption generally follows a similar pattern to the susceptibility of the starch to enzymic degradation (Evers and Stevens 1985). Figure 3C supports this view although the effect of roll gap was less significant (P < 0.05) than the effect of differential. Indeed, because of the inconsistency between large and intermediate roll gaps, roll gap may not exert an effect on flour water absorption.

Compositional change in flour is shown by ash content and color values. Roll gap had no influence on ash content (Fig. 4). In this respect the results for ash were similar to the results for flour protein content (Fig. 2). Flour ash content and protein content were closely associated because both increase from the inner to the outer part of the wheat kernel (McMasters et al 1971). Flour color was significantly related to roll gap (P < 0.001), but this

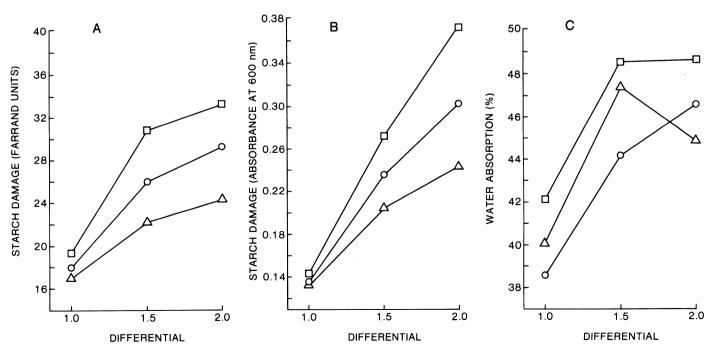


Fig. 3. Effect of roll differential at roll gaps of 63  $\mu$ m ( $\Delta$ ), 38  $\mu$ m ( $\Omega$ ), and 30  $\mu$ m ( $\square$ ) on A, starch damage determined enzymatically; B, starch damage determined by amylose extractability; and C, water absorption for flour produced during reduction of hard red spring wheat farina.

relationship was inconsistent as differential changed (Fig. 4B). Flour ash and color were both significantly (P < 0.001) affected by differential; flour ash and color decreased when differential changed from 1:1 to 1.5:1 and increased when differential increased to 2:1. The higher flour ash and darker flour color at 1:1 differential compared to the other differentials was unexpected. A previous study showed a continuous decrease in ash and color values as differential was decreased from 2:1 to 1.1:1 (Scanlon and Dexter 1986). Since increased differential should induce more tearing of the bran, one would predict greater flattening rather than tearing of bran particles at 1:1 differential. Flattened bran particles would not pass into the flour, thus at 1:1 differential lower ash content and brighter color values should result.

#### Particle-Size Related Properties

The particles making up flour range in size from fragments of starch and protein to collections of whole starch granules embedded in protein. The proportions of all fractions, obtained by sieving 100 g of each flour, were significantly (P < 0.001) related to roll gap and differential (Fig. 5).

Changes in one of the components making up these fractions as roll gap and differential were altered are shown in Figure 6. Protein

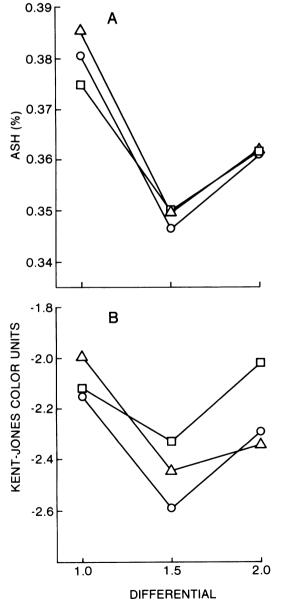


Fig. 4. Effect of roll differential at roll gaps of  $63 \,\mu\mathrm{m}$  ( $\Delta$ ),  $38 \,\mu\mathrm{m}$  ( $\omega$ ), and  $30 \,\mu\mathrm{m}$  ( $\omega$ ) on A, ash content and B, color grade of flour produced during reduction of hard red spring wheat farina.

content varied between fractions, being highest in the coarse fraction and lowest in the fine fraction. The lower protein content of the fine fraction presumably reflects the presence of individual starch granules. The wedge protein that enveloped starch granules

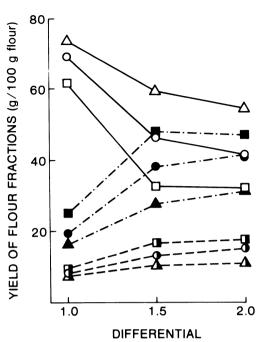


Fig. 5. Particle size distribution of flour released during smooth roll grinding of farina at three roll differentials and roll gaps of 63  $\mu$ m ( $\Delta$ ), 38  $\mu$ m ( $\sigma$ ), and 30  $\mu$ m ( $\sigma$ ). The proportion of each particle size fraction (91–136  $\mu$ m [——], 53–91  $\mu$ m [— ·—], and <53  $\mu$ m [– –—]) was determined by sifting composite flour.

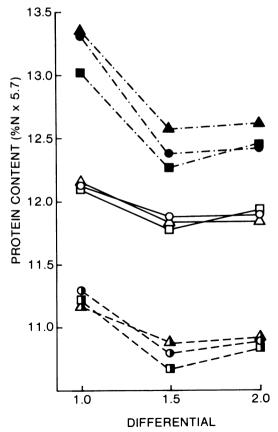


Fig. 6. Protein content of flour particle size fractions (91–136  $\mu$ m [——], 53–91  $\mu$ m [—·—], and <53  $\mu$ m [— ——]) released during smooth roll grinding of farina at three roll differentials and roll gaps of 63  $\mu$ m ( $\Delta$ ), 38  $\mu$ m ( $\Omega$ ), and 30  $\mu$ m ( $\Omega$ ).

in the intact endosperm still adhered to the granules of coarser flour particles. All flour fractions and the composite flours showed similar relationships of protein content to roll gap and differential (Figs. 2 and 6).

Flour physical properties also vary according to particle size. The lower portions of Figure 7A and B show the significant (P < 0.001) increase in both starch damage assays as differential increases and roll gap decreases for the coarse and medium fractions. Some overlap is apparent for these two fractions depending on the roll gap and differential employed, but not for the fine material which exhibited much greater starch damage values than the larger sized flour fractions. Increasing the compressive stresses by reducing the roll gap did not alter starch damage in the fine fraction (P > 0.05). However, starch damage is significantly (P < 0.001) related to the applied shear stresses imparted by roll differential.

The fine fraction also exhibited greater water absorption compared to the coarser fractions (Fig. 7C). Contrary to what would be expected, decreased roll gap decreased the water absorption (P < 0.01) of the fine fraction. For the fine fraction, increases in water absorption with increased differential followed the pattern for enzymatically determined starch damage (at a significance level of P < 0.001). However, differential had no consistent effect on the water absorption of the coarser fractions.

The decreased amount of structural integrity in the smaller fraction was confirmed by DSC. The fine fractions (Table I) exhibited reduced enthalpies ( $\Delta$ H) of starch gelatinization compared with the corresponding composite flours. The decreased  $\Delta$ H values are an indication of less crystalline organization in the starch granule (Biliaderis 1983, Stevens and Elton 1971). The significantly (P < 0.001) lower  $\Delta$ H values for the fine fractions compared to the composite flours intimates that the starch in the fine fraction was less crystalline than in the coarser fractions. Differential had no significant (P > 0.05) effect on  $\Delta$ H, whereas roll gap was related to  $\Delta$ H in an inverse relationship (P < 0.05).

## Compressive Stress and Structure

In order to explain how the various roll gaps and differentials

cause different properties in the fractions (and thus in the flour as a whole) one must consider the way in which the applied compressive and shear forces act on the structure of the farina.

In hard wheats a strong adhesion occurs between the starch granule and the protein (Barlow et al 1973). The stress from the outward growth of the starch granule and prolonged contact time at the interface allows the adsorption and interdiffusion necessary for strong adhesion (Kaelble 1971). At physiological maturity the interface is a very complex network of interdigitating starch and

TABLE I
Differential Scanning Calorimetric Characteristics of Flours
and < 53-\mu m Fractions<sup>a</sup>

		ΔH of Starch		
Sample Type	Roll Gap	Differential	Melting Transition (J/g)	Transition Peak Temperature (°C)
Flour	Large	1.0:1 1.5:1 2.0:1	$6.81 \pm 0.15$ $6.80 \pm 0.17$ $6.25 \pm 0.24$	$62.4 \pm 0.9$ $62.5 \pm 0.5$ $62.3 \pm 0.5$
	Medium	1.0:1 1.5:1 2.0:1	$5.62 \pm 0.30$ $6.16 \pm 0.06$ $5.59 \pm 0.38$	$61.9 \pm 0.4$ $62.0 \pm 0.6$ $61.7 \pm 0.3$
	Small	1.0:1 1.5:1 2.0:1	$5.62 \pm 0.15$ $5.29 \pm 0.26$ $4.63 \pm 0.25$	$61.8 \pm 0.3$ $62.0 \pm 0.4$ $61.7 \pm 0.5$
<53-μm fraction	Large	1.0:1 1.5:1 2.0:1	$4.21 \pm 0.18$ $3.62 \pm 0.29$ $3.32 \pm 0.10$	$62.7 \pm 0.1$ $62.0 \pm 0.2$ $60.3 \pm 1.6$
	Medium	1.0:1 1.5:1 2.0:1	$3.94 \pm 0.24$ $3.75 \pm 0.27$ $3.51 \pm 0.18$	$62.6 \pm 0.1$ $61.4 \pm 0.1$ $62.0 \pm 1.0$
	Small	1.0:1 1.5:1 2.0:1	$4.53 \pm 0.20$ $4.39 \pm 0.15$ $4.77 \pm 0.18$	$61.5 \pm 0.8$ $62.3 \pm 0.3$ $62.4 \pm 0.5$

a Results are averages of three replicates.

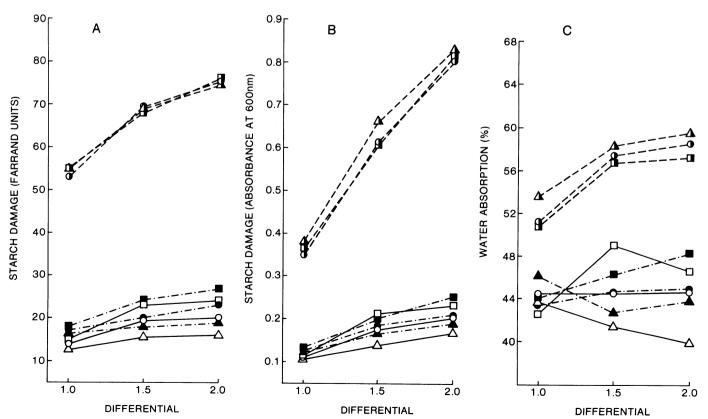


Fig. 7. Starch damage and water absorption of flour particle size fractions (91–136  $\mu$ m [——], 53–91  $\mu$ m [—·—], and <53  $\mu$ m [— ——]) released during smooth roll grinding of farina at three roll differentials and roll gaps of 63  $\mu$ m ( $\Delta$ ), 38  $\mu$ m ( $\Delta$ ), and 30  $\mu$ m ( $\Box$ ). A, Starch damage determined enzymatically; B, starch damage determined by amylose extractability; and C, water absorption.

protein molecules (and other components such as lipids (Simmonds and O'Brien 1981)) rather than a clearly defined demarcation line (Lineback 1984).

Despite the strong interface, most starch granules are released upon milling relatively intact (Williams and LeSeelleur 1970), although some tear marks can be seen (Jones 1940). Intact granules will be found almost exclusively in the fine fraction. Increasing a given state of stress will increase the likelihood of fracture whatever its mode (i.e., ductile, brittle, or mixed-mode [Berry 1972]). This was seen by the amount of flour released for a given differential (Fig. 1A) and also for the proportion of fine material produced (Fig. 5) that showed continual increase as differential increased and roll gap decreased.

A greater number of fractures will be initiated in those regions of the farina particle where stresses are greater. In a heterogeneous matrix, increased stress levels occur at the interface of the inclusion and the matrix (Davidge and Green 1968) due to the differing elastoplastic nature of the two phases. Using a micropenetrometer Barlow et al (1973) found the protein matrix within wheat endosperm to have a 20% greater modulus of elasticity than the starch granule. The stress concentration between the two phases arising from this difference, coupled with any incompletion of the bonding process between the starch granule inclusions and the protein matrix (since voids increase the stress concentration [Ko 1972]), will cause fracture initiation at the starch-protein interface.

How fractures develop depends on the concentration of starch granules and the conditions of stressing. Where there is a lower concentration of starch granules the greater amount of protein present will bind the starch granules more tightly. This increases the likelihood of fractures developing brittlely (Chamis 1974), causing fast crack formation, as outlined by Miyata and Jinno (1972). Inhomogeneities (such as starch granules) can disturb the stress field at the crack tip so crack branching can occur (Theocaris 1981). This creates greater numbers of large particles. Where the concentration of starch granules is greater, the growth of cracks formed at the interface of each granule may spread from one granule to another, eventually meeting two surfaces so that two or more new particles are formed (Miyata and Jinno 1972). Starch granules are released where the crack plane has developed around both "hemispheres" of the starch granule.

Increased compression will not change the mode of fracture. Greater stresses will increase the number of cracks initiated at the interfaces, increasing the amount of particles formed (Fig. 5). Since the mode of fracture is the same, the degree of starch damage will not be altered by increased compression. Enzymatic and amylose extractability starch-damage assays, water absorption (Fig. 7), and DSC data (Table I) show that structural damage to starch is independent of roll gap setting for the fine fraction.

# Differential and Structure

Since the particles are held on the slow roll (Scott 1951) applying a differential will cause rolling of the farina particles within the grinding zone by the fast roll, setting up stresses in different regions of the particles. For a given particle, a greater volume will thus be subjected to the compressive stresses of the rolls, generating more fractures according to the fracture process described above, creating greater numbers of particles than at 1:1 differential (Figs. 1A and 5).

A second effect of increasing differential is an almost linear increase in starch damage (Fig. 7). As the compressive stresses are causing breakage of the adhesive bonds at the interface, there will be a force acting across the interface (imparted by the differential) attempting to separate the two newly forming surfaces. During the time necessary for the separation of the strongly bound starch and protein components, stresses will be acting on the starch chains at right angles to the periphery of the surface of the granule, and parallel to the crystallite alignment. Providing the outer portions of the starch chains are still bound to the protein, crystallite slippage (initiating from defects [Guy 1976] such as the amorphous regions within the granule) can occur, since ductile fracture is more readily effected by shear stresses (Bucknall et al 1972). The loss of crystalline integrity will be seen as increased starch damage and as

lower  $\Delta H$  values of the fine-fraction DSC thermograms. If the stress is sufficient, the shear yielding that occurs within the granule may produce tear marks (Jones 1940). The greater the intensity of the force attempting to separate the forming crack, the greater the stress imposed on the starch chains at the interface, thus increasing the probability of crystallite slippage. This will account for the continued increase in starch damage in the fine fraction as differential increases.

It was previously postulated (Scanlon and Dexter 1986) that the increase in the ratio of starch damage determined by extractability to starch damage determined by  $\alpha$ -amylase digestibility as differential increased was due to more fine material in the flour. Table II shows that the extractability/digestibility ratio increases as differential is increased for all flour particle-size fractions. The mode of fracture outlined above will account for this increase. The greater the deformation imposed on the amorphous regions by the increased differential, the more they will open up and provide greater access to the extracting medium.

The increase in milling energy consumed as differential increased (Fig. 1B) is consistent with increased loss of crystallite integrity since shear yielding of the crystallite regions requires continued expenditure of energy (Guy 1976).

## **Protein Fracture**

The diversity in structure and size of wheat endosperm proteins results in inefficient packing, long molecular relaxation times, and numerous secondary bonding (Lasztity 1984). These factors have been suggested to contribute to the amorphous structure of gluten (Hoseney et al 1986). Movement of molecules relative to one another is inhibited in amorphous materials, and so they are more readily susceptible to brittle crack propagation as a result of normal stresses (Berry 1972).

Brittle fracture through gluten's glasslike structure is favored when differential is absent and starch granule concentration is lower. Upon initiation from the interface, the crack can propagate in a brittle manner through the protein matrix. The results for all sized fractions (Fig. 6) and the composite flour (Fig. 2) showed that at 1:1 differential breakage through the high-protein regions formed progeny particles of high protein. Decreasing the roll gap should not change the mode of fracture, only initiate more of the same type, due to greater compressive stresses. Thus roll gap exerted no effect on protein content, just as the mode of fracture creating starch damage in the fine fraction was unaffected by roll gap.

A similar mode of fracture will account for the anomalous result of higher ash and color values at a differential of 1:1 (Fig. 4). Brittle fracture through these proteinaceous regions may also traverse any bran adhering to this outer endosperm so that any flour-sized particles formed will have greater ash content and poorer flour color. Another possible explanation was propounded by Barnes (1986) recently. He saw that bran was not the only contributor to changes in flour color grade. Increases in protein content of the

TABLE II
Variation in Extractability/Digestibility Ratio with Particle Size

Fraction	Differential	Farrand Units <sup>a</sup>	Absorbance at 600 nm <sup>a</sup>	Extractability/ Digestibility Ratio × 100
Flour	1.0:1	17.10	0.1372	0.80
	1.5:1	22.30	0.2391	1.07
	2.0:1	24.30	0.3018	1.24
Coarse	1.0:1	14.36	0.1099	0.77
	1.5:1	19.71	0.1737	0.88
	2.0:1	20.42	0.1999	0.98
Medium	1.0:1	17.45	0.1218	0.70
	1.5:1	20.98	0.1812	0.86
	2.0:1	23.42	0.2156	0.92
Fine	1.0:1	54.33	0.3606	0.66
	1.5:1	69.47	0.6270	0.90
	2.0:1	75.87	0.8194	1.08

a Mean for three roll gaps.

wheat endosperm were related to decreases in the flour color grade. Barnes (1986) ascribed this either to differences in the concentration of light-absorbing components, or to differences in light scattering due to decreased numbers of starch granules in the higher protein regions. The greater amount of protein in the flour at a differential of 1:1 than at the higher differentials (Fig. 2) would be enough to decrease the flour color grade beyond any improvement afforded by the lack of bran tearing that occurs at 1:1 differential. The strong association of protein content and ash content within various regions of the kernel (Mc Masters et al 1971) will account for the similar increase in ash content at 1:1 differential.

#### **CONCLUSIONS**

Starch damage in hard wheat flour was found to be strongly dependent on particle size. Structural alteration by grinding was much greater in particle sizes less than 53  $\mu$ m compared with coarser fractions. Greater compressive forces did not alter the degree of starch damage in the fine fraction of the flour. This was surmised to be a consequence of greater stress causing more fracture, rather than altering the mode whereby particles were released from the parent farina particles.

Increased differential increased the starch damage as a result of shearing forces acting at the starch/protein interface causing crystallite slippage within the starch granule. The ratio of amylose extracted to starch digestibility by  $\alpha\text{-amylase}$  increased with increasing differential for all three flour fractions. The increase in extractability/digestibility ratio with increasing differential can be ascribed to greater crystallite slippage as differential increased, disrupting the amorphous regions containing the amylose.

At 1:1 differential, movement of crystallites is inhibited due to reduced shearing forces. Under these conditions brittle fracture through the more proteinaceous regions is favored. This accounts for the greater protein content, higher ash content and darker color of the flour at 1:1 differential.

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# LITERATURE CITED

- AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1983. Approved Methods of the AACC. Method 08-01, revised October 1981. The Association: St. Paul, MN.
- BARLOW, K. K., BUTTROSE, M. S., SIMMONDS, D. H., and VESK, M. 1973. The nature of the starch-protein interface in wheat endosperm. Cereal Chem. 50:443.
- BARNES, P. J. 1986. The influence of wheat endosperm on flour colour grade. J. Cereal Sci. 4:143.
- BERRY, J. P. 1972. Fracture of Polymeric Glasses. Page 37 in: Fracture: An Advanced Treatise. Vol. 7. H. Liebowitz, ed., Academic Press: New York.
- BILIADERIS, C. G. 1983. Differential scanning calorimetry in food research—A review. Food Chem. 10:239.
- BILIADERIS, C. G., PAGE, C. M., SLADE, L., and SIRET, R. R. 1985. Thermal behavior of amylose-lipid complexes. Carbohydr. Polym. 5:367.
- BLACK, H. C., HSIEH, F-.H., MARTIN, D. G., and TIPPLES, K. H. 1980. Two Grain Research Laboratory research mills and a comparison with the Allis-Chalmers mill. Cereal Chem. 57:402.
- BUCKNALL, C. B., GOTHAM, K. V., and VINCENT, P. I. 1972. Fracture, an empirical approach. Page 261 in: Polymer Science. Vol. 1. A. D. Jenkins, ed. North Holland Publishing Co.: Amsterdam.
- BUSHUK, W., and WRIGLEY, C. W. 1974. Proteins: Composition, structure and function. Page 119 in: Wheat: Production and Utilization.
   G. E. Inglett, ed. Avi Publishing Co.: Westport, CT.
- CHAMIS, C. C. 1974. Mechanics of load transfer at the interface. Page 31

- in: Composite Materials. Vol. 6. Interfaces in Polymer Matrix Composites. E. P. Plueddemann, ed. Academic Press: New York.
- DAVIDGE, R. W., and GREEN, T. J. 1968. The strength of two-phase ceramic/glass materials. J. Mater. Sci. 3:629.
- DUNCAN, D. B. 1955. Multiple range and multiple F test. Biometrics 11:1.
  EVERS, A. D., and STEVENS, D. J. 1985. Starch Damage. Page 321 in: Advances in Cereal Science and Technology. Vol. 7. Y. Pomeranz, ed. Am. Assoc. Cereal Chem.: St. Paul, MN.
- EVERS, A. D., BAKER, G. J., and STEVENS, D. J. 1984. Production and measurement of starch damage in flour. I. Damage due to roller milling of semolina. Staerke 36:309.
- FARRAND, E. A. 1964. Flour properties in relation to the modern bread process in the United Kingdom, with special reference to  $\alpha$ -amylase and starch damage. Cereal Chem. 41:98.
- GUY, A. G. 1976. Essentials of Materials Science. McGraw-Hill Book Company: New York.
- HOLAS, J., and TIPPLES, K. H. 1978. Factors affecting farinograph and baking absorption. I. Quality characteristics of flour streams. Cereal Chem. 55:637.
- HOOPER, J. A. 1971. The failure of glass cylinders in diametral compression. J. Mech. Phys. Solids 19:179.
- HOSENEY, R. C. 1986. Principles of Cereal Science and Technology. The American Association of Cereal Chemists: St. Paul, MN.
- HOSENEY, R. C., ZELEZNAK, K., and LAI, C. S. 1986. Wheat gluten: A glassy polymer. Cereal Chem. 63:285.
- JONES, C. R. 1940. The production of mechanically damaged starch in milling as a governing factor in the diastatic activity of flour. Cereal Chem. 17:133.
- KAELBLE, D. H. 1971. Physical Chemistry of Adhesion. Wiley Interscience: New York.
- KILBORN, K. H., BLACK, H. C., DEXTER, J. E., and MARTIN, D. G. 1982. Energy consumption during flour milling: Description of two measuring systems and the influence of wheat hardness on energy requirements. Cereal Chem. 59:284.
- KO, K. C. 1972. Elastic stresses in two-phase composites. Page 19 in: Mechanical Behavior of Materials. Vol. 5. The Society of Materials Science: Kyoto, Japan.
- KOZMIN, P. A. 1921. Grinding the grain. Page 153 in: Flour Milling. George Routledge and Sons, Ltd.: London.
- LASZTITY, R. 1984. The Chemistry of Cereal Proteins. CRC Press: Boca Raton. FL.
- LELIEVRE, J. 1974. Starch damage. Staerke 26:85.
- LINEBACK, D. R. 1984. The starch granule: Organization and properties. Bakers Dig. 58(2):16. 16.
- McDERMOTT, E. E. 1980. The rapid non-enzymatic determination of damaged starch in flour. J. Sci. Food Agric. 31:405.
- McMASTERS, M. M., HINTON, J. J. C., and BRADBURY, D. 1971.
  Microscopic structure and composition of the wheat kernel. Page 51 in:
  Wheat Chemistry and Technology, 2nd ed. Y. Pomeranz, ed. Am. Assoc.
  Cereal Chem.: St. Paul, MN.
- MIYATA, N., and JINNO, H. 1972. Fracture of two-phase glass-crystal composites. J. Mater. Sci. 7:973.
- PRESTON, K. R., and TIPPLES, K. H. 1978. An ultracentrifuge flour absorption method. Cereal Chem. 55:96.
- SCANLON, M. G., and DEXTER, J. E. 1986. Effect of smooth roll grinding conditions on reduction of hard red spring wheat farina. Cereal Chem. 63:431.
- SCOTT, J. H. 1951 Flour Milling and Processes, 2nd ed. Chapman and Hall: London. 316 p.
- SIMMONDS, D. H., and O'BRIEN, T. P. 1981. Morphological and biochemical development of the wheat endosperm. Page 5 in: Advances in Cereal Science and Technology. Vol. 4. Y. Pomeranz, ed. Am. Assoc. Cereal Chem.: St. Paul, MN.
- SNEDECOR, G. W., and COCHRAN, W. G. 1967. Statistical Methods. Iowa State University Press: Ames, IA.
- STEVENS, D. J., and ELTON, G. A. H. 1971. Thermal properties of the starch/water system. Staerke 23:8.
- THEOCARIS, P. S. 1981. Secondary afterfailure fractures due to transversely reflected waves. Eng. Fract. Mech. 15:283.
- WILLIAMS, P. C. 1970. The relationship of flour particle size and damaged starch content to the industrial usage of flour. Page 6/67 in: Proc. Int. Bread Wheat Flour Congr., 5th, Dresden, DDR. ICC: Vienna, Austria.
- WILLIAMS, P. C. 1973. The use of titanium dioxide as a catalyst for large scale determination of the total nitrogen content of cereal grains. J. Sci. Food Agric. 24:343.
- WILLIAMS, P. C., and LESEELLEUR, G. C. 1970. Determination of damaged starch in flour. Cereal Sci. Today 15:4.