

# Collaborative Analysis of Wheat Endosperm Compressive Material Properties

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## ABSTRACT

Cereal Chem. 88(4):391–396

The objective measurement of cereal endosperm texture, for wheat (*Triticum* spp. L.) in particular, is relevant to the milling, processing, and utilization of grain. The objective of this study was to evaluate the interlaboratory results of compression failure testing of wheat endosperm specimens of defined geometry. Parallelepipeds (bricks) and cylinders were prepared from individual soft and hard near-isogenic wheat kernels and compressed in two orientations (parallel and perpendicular to the long brush-to-germ axis). Compression curves were used to derive failure stress, failure strain, work density (area under the curve), and Young's modulus. In all five laboratories, the ability to delineate hard from soft wheat endosperm material properties was quite high. Four laboratories compressed endosperm bricks in the same orientation, on edge; texture class (soft vs. hard) was consistently the greatest source of variation in analysis of variance models (*F*-values from 417 to 1401, Young's modulus and failure stress, respectively). Failure stress was found to be the best overall means of measuring the difference in what is known in the vernacular as wheat hardness. Across laboratories, the absolute

measures of all four material properties ranged on the order of about two- to threefold from low to high, although within a laboratory, results were highly consistent. Laboratory by texture class interaction was deemed to be of minor importance. Brick size and moisture content within the ranges tested were not major sources of variation, and cylinders prepared from endosperm produced results similar to those obtained from bricks. The results suggested that wheat endosperm might express some level of anisotropic behavior, as specimens compressed in the kernel orientation parallel to the long axis failed at lower strain and stress values, with lower work density, when compared with kernel orientation perpendicular to the long axis. A key feature of interlaboratory variation was identified as being instrument rigidity, a subject of ongoing research. In conclusion, the preparation of endosperm specimens of defined size and shape, in combination with compression failure testing at low moisture content (<18%), is useful for objectively delineating the phenomenon known as hardness. The study presented here will advance our ability to objectively measure cereal grain texture and the material properties of endosperm.

The hardness or texture of cereal grains is an important feature of how they are processed for foods and feeds, and it imparts various properties to the resulting meals and flours, such as particle size distribution and starch damage. Wheat (*Triticum* spp. L.) is unique in that there are three defined hardness classes: soft and hard hexaploid wheat (*T. aestivum*) and very hard durum wheat (*T. turgidum* subsp. *durum*) (Morris 2002; Bhavane and Morris 2008). Although there are a number of suitable methods to measure the texture of wheat grains empirically (Morris 2002; Bhavane and Morris 2008), none provide absolute measures of true material properties. The primary limitation of these methods relates to the complex geometry of the wheat kernel itself (Shpolyanskaya 1952; Kozma and Cunningham 1962; Shelef and Mohsenin 1967; Arnold and Roberts 1969; Multon et al 1981; Al Saleh and Gallant 1985; Glenn and Johnston 1992; Dobraszczyk 1994; Kang et al 1995; Ponce-García et al 2008; Elbatawi 2009; Figueroa et al 2009). Glenn et al (1991) were the first to surmount this limitation by providing a means of producing specimens of wheat endosperm of defined geometry free of bran and germ tissues. Subsequent researchers employed this strategy, either by producing cylinders or by producing rectangular parallelepipeds

(bricks) (Glenn et al 1991; Jolly et al 1996; Haddad et al 1998, 1999, 2001; Delwiche 2000; Osborne et al 2001, 2007; Dobraszczyk et al 2002; Samson et al 2005; Greffeuille et al 2006, 2007; Morris et al 2007, 2008a, 2008b; Wang and Jeronimidis 2008).

The utility of endosperm texture analysis of wheat and other cereals is clear; however, the methods described previously for producing and testing cylinders or bricks have not yet been sufficiently generalized for widespread use. Here we report on a multi-laboratory collaborative trial using a common source of wheat grains of defined texture genetics. Differences among laboratories resulting from testing protocols are indicated, endosperm bricks vs. cylinders are compared, and the orientation of the specimen during compression is examined. Results are discussed in the context of specimen size, shape, and orientation. Instrument rigidity (compliance) is identified as an important parameter for normalizing apparent material properties and is considered more fully in forthcoming research.

## MATERIALS AND METHODS

Participating laboratories included the U.S. Department of Agriculture, Agricultural Research Service (USDA-ARS) Western Wheat Quality Laboratory (WWQL), USDA-ARS Food Quality Laboratory (FQL), USDA-ARS Center for Grain and Animal Health Research (CGAHR), Washington State University (WSU) Department of Biological Systems Engineering, and the Institut National de la Recherche Agronomique (INRA).

Wheat kernels were from soft and hard near-isogenic lines derived from the cultivar Alpowa (soft, PI 566596; hard, PI 644080) (Morris and King 2008) grown together under glasshouse conditions. Endosperm bricks were prepared at WWQL by the method of Morris et al (2007). Dimensions of individual bricks were recorded and were approximately 2.1 × 0.9 × 0.7 mm (Morris et al 2008a, their Fig. 1B). The longest dimension corresponds to the brush-to-germ axis of the kernel; the brick is derived from approximately the midportion of the cheek. Sixteen soft and 16 hard endosperm bricks were sent to each collaborating laboratory via express courier in individual 1.7-mL microcentrifuge tubes. Six practice bricks were also included in each shipment; these data were not used in any of the analyses presented here.

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Additionally, whole kernels (six from hard and 18 from soft kernels) were sent to FQL for the lathing of cylinders according to the procedure of Glenn et al (1991). Dimensions were approximately 1 mm in diameter  $\times$  3 mm long plus 0.5 mm of unturned (but surface-faced to beyond the germ) base at the proximal end. The axis of the cylinder coincided with the long axis (brush to germ) of the wheat kernel. Cylinders were compressed parallel to this axis. The cylinders from the soft kernels were subdivided, with six tested using the common (0.25 mm/sec) compression rate and 12 compressed at a slower (0.00833 mm/sec) rate following Delwiche (2000). Further, the six hard cylinders were compressed at 0.00833 mm/sec.

Whole kernels were also sent to INRA, where additional bricks (six soft and six hard) were prepared according to the procedure of Haddad et al (1998). Dimensions were 2.5–3.0  $\times$  1.3–1.6  $\times$  0.8–1.0 mm. All bricks tested at INRA were equilibrated to 15% moisture content over saturated NaCl solution. In lieu of using a small trigger threshold, the force vs. displacement readings were manually inspected at the end of each test to determine the zero strain mark coincident with brick contact.

### Compression Analysis of Endosperm Bricks and Cylinders

FQL used an Instron model 4464 with Merlin version 9 software, a load cell of 500 N, and a data collection rate of 20 points/sec. All bricks and some cylinders, as noted previously, were compressed with a crosshead speed of 0.25 mm/sec; some cylinders were compressed at a rate of 0.00833 mm/sec. The crosshead stopped at the first occurrence of any of the following: 90% strain (using a 0.2-N threshold to sense contact with the brick), 1.95 mm total travel (plate separation distance was 2.00 mm), or 450 N on load cell. Bricks were compressed sitting on the length  $\times$  width axis face (Morris et al 2008a, their Fig. 1B) (on edge, wherein the brush-to-germ axis is perpendicular to the applied force). Cylinders were loaded on end (brush-to-germ axis parallel to applied force).

CGAHR used an Instron model 4400. The transducer, or load cell, was a model 2550-416 with a 135.6-N·m (100-lbf·ft) capacity. The crosshead speed was 0.25 mm/sec. Bricks were tested on edge.

WSU tested kernel bricks using a hydraulic-powered Instron model 1350 with a model 2580 load cell with 500-N capacity. The loading rate was 0.485 mm/sec. Deformation and force were recorded at a rate of 0.005 mm/sec using a Labview data collection routine (Pitts unpublished). Bricks were tested on edge.

WWQL used a TA.XTPlus texture analyzer (Texture Technologies, Scarsdale, NY, and Stable Micro Systems, Godalming, UK) running Exponent software and equipped with a 50-kg load cell, a 10-mm (diameter) stainless steel probe, and a stainless steel base plate. The crosshead speed was 0.25 mm/sec with an upper strain limit of 90% and a 0.2-N trigger force. Bricks were compressed on edge. Compression data curves were acquired at 200 points/sec and used to calculate all other stress vs. strain material properties. These and all other procedures followed those detailed by Morris et al (2008a, 2008b). Although not measured, the ambient equilibrium moisture content of grain (and bricks) at WWQL is on the order of 9–10%.

INRA used a Zwick instrument (Zwick, Ulm, Germany), a single-column testing machine for tensile, compression, and cyclic testing with a load cell of 500 N. The endosperm bricks were compressed on end, with a speed rate of 0.5 mm/sec. This device is fully compatible with testXpert version 6.01 software (Zwick).

Data analysis (analysis of variance [ANOVA] in general linear models) was performed using SAS statistical software (v. 9.0, SAS Institute, Cary, NC). Mean separation was assessed using Duncan's multiple range test with  $\alpha = 0.05$ . Type III mean squares and *F*-tests are reported.

## RESULTS

### Compression Analysis of Endosperm Bricks Perpendicular to the Kernel's Longitudinal Axis

Four laboratories (FQL, CGAHR, WWQL, and WSU) analyzed the 16 soft and 16 hard endosperm bricks prepared at WWQL in the on-edge orientation, that is, compression perpendicular to the long axis (which corresponds to the brush-to-germ axis) of the kernel, with the brick sitting on its narrower side (Morris et al 2008a, their Fig. 1B) (Table I). The four response variables were strain at maximum stress, maximum stress, the work per unit volume of endosperm up to the point of maximum stress, and Young's modulus. (Hereafter, the first three variables will be referred to as failure strain, failure stress, and work density, respectively.)

Two-way ANOVA (laboratory and kernel texture class with interaction term) returned robust models with  $R^2$  of 0.92–0.94 for failure strain, failure stress, and Young's modulus and 0.85 for work density (Table II). Of the four measures, failure stress

TABLE I  
Compressive Strength of Endosperm Specimens Produced from Near-Isogenic Hard and Soft Wheat Kernels

Laboratory <sup>a</sup>	Texture Class	Specimen	Orientation <sup>b</sup>	<i>n</i>	Failure Strain (%)	Failure Stress (MPa)	Work Density (MJ/m <sup>3</sup> )	Young's Modulus (GPa)
FQL	Hard	Brick	Edge	16	14.28 $\pm$ 2.15	106.0 $\pm$ 9.2	7.13 $\pm$ 1.93	1.59 $\pm$ 0.23
FQL	Soft	Brick	Edge	16	7.47 $\pm$ 1.37	29.9 $\pm$ 5.6	0.85 $\pm$ 0.24	0.69 $\pm$ 0.09
FQL	Hard	Cylinder <sup>c</sup>	End	6	5.29 $\pm$ 1.00	59.6 $\pm$ 9.3	2.00 $\pm$ 0.63	1.88 $\pm$ 0.22
FQL	Soft	Cylinder <sup>c</sup>	End	12	2.29 $\pm$ 0.31	25.2 $\pm$ 3.2	0.35 $\pm$ 0.08	1.49 $\pm$ 0.17
FQL	Soft	Cylinder	End	6	2.58 $\pm$ 0.20	25.1 $\pm$ 1.7	0.39 $\pm$ 0.04	1.22 $\pm$ 0.09
CGAHR	Hard	Brick	Edge	16	22.15 $\pm$ 4.77	95.7 $\pm$ 23.2	12.12 $\pm$ 5.06	0.47 $\pm$ 0.06
CGAHR	Soft	Brick	Edge	16	8.46 $\pm$ 1.48	25.6 $\pm$ 6.5	1.38 $\pm$ 0.39	0.34 $\pm$ 0.08
WSU	Hard	Brick	Edge	16	13.49 $\pm$ 1.70	116.2 $\pm$ 11.0	7.60 $\pm$ 2.00	1.61 $\pm$ 0.29
WSU	Soft	Brick	Edge	16	10.65 $\pm$ 1.78	32.0 $\pm$ 5.6	2.45 $\pm$ 2.40	0.51 $\pm$ 0.16
WWQL	Hard	Brick	Edge	16	48.80 $\pm$ 6.89	94.7 $\pm$ 11.3	24.19 $\pm$ 6.32	0.22 $\pm$ 0.02
WWQL	Soft	Brick	Edge	16	17.85 $\pm$ 2.33	27.6 $\pm$ 4.3	2.84 $\pm$ 0.76	0.15 $\pm$ 0.02
INRA	Hard	Brick	End	16	7.36 $\pm$ 1.81	28.6 $\pm$ 8.0	1.32 $\pm$ 0.58	0.55 $\pm$ 0.18
INRA	Soft	Brick	End	16	6.70 $\pm$ 1.61	10.7 $\pm$ 2.7	0.45 $\pm$ 0.18	0.25 $\pm$ 0.08
INRA	Hard	Brick <sup>d</sup>	End	6	7.87 $\pm$ 1.62	36.0 $\pm$ 6.1	1.84 $\pm$ 0.05	0.79 $\pm$ 0.32
INRA	Soft	Brick <sup>d</sup>	End	6	4.63 $\pm$ 0.91	14.9 $\pm$ 2.7	0.41 $\pm$ 0.09	0.42 $\pm$ 0.12

<sup>a</sup> FQL = U.S. Department of Agriculture, Agricultural Research Service (USDA-ARS) Food Quality Laboratory; CGAHR = USDA-ARS Center for Grain and Animal Health Research; WSU = Washington State University; WWQL = USDA-ARS Western Wheat Quality Laboratory; and INRA = Institut National de la Recherche Agronomique.

<sup>b</sup> Indicates the surface area that was in contact with the compression stage and probe.

<sup>c</sup> Compressed at 0.00833 mm/sec; all other cylinders were compressed at a rate of 0.25 mm/sec.

<sup>d</sup> Prepared according to the method of Haddad et al (1998).

showed the greatest effect resulting from kernel texture with much smaller effects resulting from laboratory and from laboratory by texture class interaction. For all measures, however, texture class was the most significant source of variation (Table II).

For bricks compressed on edge, mean failure strain across the two texture classes was greater for WWQL than for the other three laboratories (Table III); it was more than twice that of CGAHR and three times that of FQL. Mean failure strain from WWQL and CGAHR differed significantly from all other laboratories; WSU and FQL produced the smallest values, which were not significantly different. Mean failure stress was not different between WWQL and CGAHR (the two lowest values), whereas the higher value from FQL and highest value from WSU were different from all other laboratories (Table III). Mean work density followed in the same rank and in the general magnitude of the mean failure strain (Table III). Finally, Young's modulus was lowest from WWQL, followed by CGAHR, WSU, and FQL; again WSU and FQL had the highest values, which were not significantly different, whereas WWQL and CGAHR were different from all other laboratories (Table III). Overall, agreement among laboratories depended on the measure.

Across the four laboratories that conducted brick compression on edge, the compressive measures for the soft and hard kernel endosperms, respectively, were failure strain, 11.1 and 24.7%; failure stress, 28.8 and 103.1 MPa; work density, 1.88 and 12.76 MJ/m<sup>3</sup>; and Young's modulus, 0.421 and 0.972 GPa. In all cases, the soft and hard endosperm texture results were highly significantly different ( $P < 0.0001$ ).

#### Compression Analysis of Endosperm Bricks Parallel to the Kernel's Longitudinal Axis

The INRA laboratory conducted compression testing parallel to the long axis (i.e., with the brick on end) (Table I). Bricks prepared at WWQL (16 soft and 16 hard) produced 13 and 14 usable compression failure curves, soft and hard, respectively. These data were added to the preceding dataset and ANOVA was conducted. In general, overall model fits were quite similar (data not shown). Examination of the model components (main effects and interaction) showed that for failure strain and work density, the results were nearly identical (data not shown). For failure stress, the laboratory and interaction model component mean squares increased by about an order of magnitude, as did their respective  $F$ -values (117 and 43, laboratory and interaction, respectively) (cf. Table II). This result illustrates the effect of the lower values obtained at INRA (Table III). The results for work density showed a small increase in laboratory and interaction model components (data not shown). The model fit for Young's modulus, however, decreased slightly ( $R^2 = 0.90$ ), with an  $F$ -value of 143. For this measure, the laboratory model component decreased ( $F = 268$ ) but not nearly as much as the texture class ( $F = 42$ ). Examination of the least squares means for INRA (Table III) indicated that failure strain, failure stress, work density, and Young's modulus were all lower. INRA

also prepared bricks using the method of Haddad et al (1998) (six soft and six hard) from the same grain lots of soft and hard near-isogenic lines. These bricks were also tested in compression parallel to the long axis. Means across texture classes are presented in Table III. With the Haddad bricks, the failure strain, failure stress, and work density values were similar to those obtained on bricks prepared at WWQL. Young's modulus increased about 50%.

#### Compression Analysis of Endosperm Cylinders Parallel to the Kernel's Longitudinal Axis

FQL prepared turned (lathed) cylinders (Glenn et al 1991; Delwiche 2000) from the same lots of soft and hard wheat kernels used throughout the study and subjected them to compression testing parallel to the longitudinal axis (equivalent to INRA testing bricks on end). ANOVA was conducted on the resulting dataset using three kernel texture-compression rate treatments: hard-slow, soft-slow, and soft-fast. Model fits ranged from  $R^2 = 0.69$  for Young's modulus to 0.90 for failure strain, with  $F$ -values of 23 to 100 (data not shown). Treatment means are presented in Table IV.

Compression rate (crosshead speed) at the two rates tested (0.00833 and 0.25 mm/sec) had no significant effect on failure strain, failure stress, or work density for the soft wheat endosperm cylinders. Young's modulus, however, decreased at the higher compression rate. At the slower compression rate, soft and hard wheat cylinders were significantly different for all measures (Table IV).

Comparison of cylinder results with brick results (Tables III and IV) indicated that failure strain, failure stress, work density,

**TABLE III**  
Material Properties of Wheat Endosperm Bricks Determined by Compression Testing Perpendicular and Parallel to the Long Axis Illustrating Differences Among Laboratories and Methodologies<sup>a</sup>

Laboratory <sup>b</sup>	Failure Strain (%)	Failure Stress (MPa)	Work Density (MJ/m <sup>3</sup> )	Young's Modulus (GPa)
WWQL	33.3a	61.2c	13.5a	0.19c
CGAHR	15.3b	60.6c	6.8b	0.40b
WSU	12.1c	74.1a	5.0c	1.06a
FQL	10.9c	68.0b	4.0c	1.13a
INRA <sup>c</sup>	6.7	19.6	0.37	0.40
INRA <sup>d</sup>	6.3	25.5	0.30	0.60

<sup>a</sup> Means across soft and hard texture classes. Institut National de la Recherche Agronomique (INRA) compressed parallel to the long axis of the kernel and the other laboratories perpendicular. Values in each column followed by different letters are significantly different ( $P < 0.05$ ).

<sup>b</sup> WWQL = U.S. Department of Agriculture, Agricultural Research Service (USDA-ARS) Western Wheat Quality Laboratory; CGAHR = USDA-ARS Center for Grain and Animal Health Research; WSU = Washington State University; and FQL = USDA-ARS Food Quality Laboratory;.

<sup>c</sup> Bricks prepared at WWQL.

<sup>d</sup> Bricks prepared according to the method of Haddad et al (1998).

**TABLE II**  
Analysis of Variance for Wheat Endosperm Brick Compression Testing with Compression Perpendicular to Long Axis of the Kernel

Source	df	Failure Strain			Failure Stress			Work Density			Young's Modulus		
		Mean Square	$F$ -value	$R^2$	Mean Square	$F$ -value	$R^2$	Mean Square	$F$ -value	$R^2$	Mean Square	$F$ -value	$R^2$
Model	7	2870	253	0.94	25830	206	0.92	977	97	0.85	5.379	233	0.93
Error	120	11	...	...	125	...	...	10	...	...	0.023	...	...
Laboratory (L) <sup>a</sup>	3	3416	301	...	1292	10	...	574	57	...	7.082	307	...
Texture class (T) <sup>b</sup>	1	5843	515	...	175560	1401	...	3755	373	...	9.618	417	...
L × T	3	1207	106	...	453	4	...	426	42	...	2.225	96	...

<sup>a</sup> Four laboratories conducted compression in this orientation: U.S. Department of Agriculture, Agricultural Research Service (USDA-ARS) Food Quality Laboratory, USDA-ARS Center for Grain and Animal Health Research, Washington State University, and USDA-ARS Western Wheat Quality Laboratory.

<sup>b</sup> Bricks were prepared from soft and hard texture classes.

and Young's modulus were within the ranges observed with the brick testing. Values tended to be generally more similar to the INRA and FQL brick results.

### Coefficients of Variation Among Laboratories and Measurements

On average, across all laboratories, texture classes, specimen types, and crush orientations, the measures of failure stress, failure strain, and Young's modulus had similar coefficients of variation (CV) (17.1–19.6) (Table V). Of the three, the ANOVA indicated that failure stress was by far the most powerful measure to differentiate endosperm texture classes ( $F = 1401$ ), with a relatively small effect resulting from laboratory and an even smaller (relative) interaction term (Table II). The CVs for work density, averaging 33.1, were on the order of twofold larger than the CVs for the other three measures (Table V), whereas work density had the poorest overall model fit (Table II) as a consequence of the combination of two independent variables.

Examination of the four laboratories that conducted compression testing on bricks in the same orientation (Table III) showed that all four had similar CVs for failure strain (Table V), whereas work density and Young's modulus were more variable. The INRA CVs indicated that they were, in general, similar to or somewhat greater than the other laboratories, suggesting that on-end brick testing may introduce greater variability. The cylinders lathed and tested at FQL produced the smallest CVs overall, with similar relative relationships among the four measurements.

**TABLE IV**  
Material Properties (Means) of Wheat Endosperm Cylinders Determined by Compression Testing Parallel to the Long Axis of the Kernel at Two Compression Rates<sup>a</sup>

Compression Rate (mm/sec)	Texture Class (n)	Failure Strain (%)	Failure Stress (MPa)	Work Density (MJ/m <sup>3</sup> )	Young's Modulus (GPa)
0.00833	Hard (6)	5.29a	59.6a	2.00a	1.88a
0.00833	Soft (12)	2.29b	25.2b	0.35b	1.48b
0.25	Soft (6)	2.58b	25.1b	0.39b	1.22c

<sup>a</sup> Performed at the U.S. Department of Agriculture, Agricultural Research Service Food Quality Laboratory. Values in each column followed by different letters are significantly different ( $P < 0.05$ ).

## DISCUSSION

The results of the studies presented herein indicate that differences in the material properties of soft and hard wheat kernel endosperm can be repeatedly measured within a laboratory using specimens of defined geometry and compression failure analysis (Table II). However, the same or nearly the same absolute values for failure stress, failure strain, work density, and Young's modulus among the various laboratories were not consistently observed. Values for these measures among the four laboratories crushing endosperm bricks prepared in a single laboratory (WWQL) and crushed in the same orientation varied at times as much as sixfold. In this regard, failure stress (Table III) was shown to be the most consistent measure of texture and Young's modulus the poorest. This result indicates that factor(s) peculiar to each laboratory were influencing texture measurements. Nevertheless, the study clearly confirmed that when using geometrically defined specimens, the biological phenomenon known as wheat hardness can be accurately discriminated using compression failure mechanics.

The results in Table I indicate that one potential source of variation is the instrument used, because four of the laboratories all crushed bricks prepared at WWQL (by a single technician), bricks were of similar size, and they were crushed in the same kernel orientation. A possible effect, it could be argued, is that the environment of each laboratory (temperature and relative humidity) could have also contributed to interlaboratory variability. Eckhoff et al (1988), Glenn et al (1991), Kang et al (1995), Delwiche (2000), Morris et al (2008a, 2008b), and Elbatawi (2009) found a material response of wheat endosperm to moisture content, generally a weakening in strength with increasing moisture. However, small differences ( $\pm 1$  percentage point) in moisture content would not greatly influence failure mechanics (Delwiche 2000). In the INRA laboratory, all specimens were equilibrated to 15% moisture, a level rarely encountered in storage but of potential relevance to milling. Kang et al (1995) reported that failure stress decreased with moisture content. This factor, admittedly, was confounded with laboratory, including equipment and specimen orientation. This consideration and others are discussed further.

### Rigidity of Instruments

Instrument rigidity is a consideration in compression testing and can be examined in various components: load cell deforma-

**TABLE V**  
Coefficients of Variation for Compressive Strength of Endosperm Specimens Produced from Near-Isogenic Hard and Soft Wheat Kernels

Laboratory <sup>a</sup>	Texture Class	Specimen	Orientation <sup>b</sup>	n	Failure Strain (%)	Failure Stress (MPa)	Work Density (MJ/m <sup>3</sup> )	Young's Modulus (GPa)
FQL	Hard	Brick	Edge	16	15.0	8.7	27.1	14.6
FQL	Soft	Brick	Edge	16	18.4	18.8	28.6	12.5
FQL	Hard	Cylinder <sup>c</sup>	End	6	19.0	15.7	31.4	11.6
FQL	Soft	Cylinder <sup>c</sup>	End	12	13.7	12.7	21.9	11.5
FQL	Soft	Cylinder	End	6	7.8	6.8	11.3	7.3
CGAHR	Hard	Brick	Edge	16	21.5	24.2	41.7	11.8
CGAHR	Soft	Brick	Edge	16	17.5	25.6	28.2	23.5
WSU	Hard	Brick	Edge	16	12.6	9.5	26.3	18.2
WSU	Soft	Brick	Edge	16	16.7	17.7	98.1	31.1
WWQL	Hard	Brick	Edge	16	14.1	11.9	26.1	9.9
WWQL	Soft	Brick	Edge	16	13.0	15.5	26.8	10.4
INRA	Hard	Brick	End	14	24.6	28.0	43.9	32.1
INRA	Soft	Brick	End	13	24.0	25.5	39.7	31.0
INRA	Hard	Brick <sup>d</sup>	End	6	20.5	16.9	24.6	40.2
INRA	Soft	Brick <sup>d</sup>	End	6	19.6	18.4	21.1	27.9
Means	...	...	...	...	17.2	17.1	33.1	19.6

<sup>a</sup> FQL = U.S. Department of Agriculture, Agricultural Research Service (USDA-ARS) Food Quality Laboratory; CGAHR = USDA-ARS Center for Grain and Animal Health Research; WSU = Washington State University; WWQL = USDA-ARS Western Wheat Quality Laboratory; and INRA = Institut National de la Recherche Agronomique.

<sup>b</sup> Indicates the surface area that was in contact with the compression stage and probe.

<sup>c</sup> Compressed at 0.00833 mm/sec; all other cylinders were compressed at a rate of 0.25 mm/sec.

<sup>d</sup> Prepared according to the method of Haddad et al (1998).

tion, frame flexure, and powertrain lash, with the latter two components lumped together as instrument rigidity. All load cells operate by measuring a small deflection during the accumulation of a force on the load cell undergoing compression. In most cases, unless thin films are being measured, this source of variation is ignored (Marc I. Johnson, Texture Technologies, *personal communication*). Generally, results are reported on an as-is basis, as they are here. In the case of Texture Technologies TA-XTi instruments, the maximum load cell deflection is 400  $\mu\text{m}$  at maximum load. The other components may contribute 100–150  $\mu\text{m}$  additional deflection at maximum load. Therefore, for example, a 20-kg load is 40% on a 50-kg load cell and creates an added displacement of  $(20 \text{ kg}/50 \text{ kg}) \times (400 \mu\text{m} + 100 \mu\text{m}) = 200 \mu\text{m}$ . For a sample 0.9 mm high, 200  $\mu\text{m}$  represents a large portion of the sample's results. This deflection will result in overestimations of strain and work and an underestimation of Young's modulus. The aspect of instrument rigidity on small specimens is the subject of ongoing research by four of the authors.

### Size, Shape, and Orientation of Specimens

The INRA results indicate that the size of specimens may not be highly important, as the slightly larger bricks prepared by the method of Haddad et al (1998) produced similar values of failure strain, failure stress, and work density compared with WWQL bricks; Young's modulus was of similar magnitude but 50% greater for Haddad bricks vs. WWQL bricks (Table III). The INRA-prepared bricks were designed to obtain specimens as large as possible. Lathed cylinders, when compared with the INRA data and FQL brick data, provided values that were generally similar. (N.B.: INRA and cylinder data were both obtained by compression in the brush-to-germ axis [Table I].) Cylinders, on average, produced the lowest failure strain values and produced failure stress, work density, and Young's modulus values that were intermediate between FQL bricks and INRA bricks. There are theoretical arguments for selecting kernel orientation for compression. Although not dramatically different, the bricks crushed at FQL in the on-edge orientation (perpendicular to brush-to-germ axis) produced results that were similar to the cylinders crushed in the same laboratory but in the other (parallel) orientation (Tables III and IV). However, the data do indicate some modest amount of anisotropy does exist in wheat endosperm and is therefore a topic for further study. Within the limits tested, compression rate over a 30-fold range had negligible effects on measuring material properties. One may also consider, again, the relevance of testing from a material property or theoretical standpoint and one devoted to flour milling.

### ACKNOWLEDGMENTS

Emma Hawkins and Erin Laraway conducted the tests at CGAHR and FQL, respectively. Dan Ramseyer with WWQL produced the bricks used in this study. The financial support of CIEE (Council on International Educational Exchange) to Camille Deroo to conduct an internship at WWQL is gratefully acknowledged.

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[Received March 22, 2011. Accepted May 23, 2011.]