

AN ENERGY APPROACH TO THE CHARACTERIZATION OF THE FRACTURE RESISTANCE OF LEATHER*

ABSTRACT

The fracture resistance of chrome-tanned bovine hides was quantitatively characterized by measuring the total energy required to break the leather. This physical quantity named fracture energy is observed to more truthfully represent the fracture resistance of leather than the tensile strength or breaking elongation. Three major independent variables, moisture content, strain rate, and sampling angle were arranged according to Box-Hunter's experimental design matrix, thereby deriving a second order polynomial equation. The statistical model so obtained concisely expressed the relationship between the variables and corresponding fracture resistance. The results showed that water acting as a plasticizer enhances the fracture resistance of leather. However, fracture energy started to decrease once the moisture content increased to around 90%. Contrary to its effect on tensile strength, the sampling angle has shown little effect on the fracture energy of leather. The effect of strain rates on fracture energy is not straightforward. The fracture energy at first decreases then increases with increasing strain rates. The ratio of tensile strength of Young's modulus was used to represent the toughness of leather. This parameter is dimensionless, independent of the geometric shape of the leather samples. A correlation was observed between this parameter and fracture energy.

INTRODUCTION

The fracture resistance of any material is of course an important factor in determining the end uses for which the substance will be suitable. There are many different measures of the fracture resistance of leather. Probably the most commonly used measurement is the one called tensile strength, which normally determines the load required to fracture a test specimen under a constant strain rate.¹ Besides tensile strength, breaking elongation is also used to characterize the fracture resistance.² Leather is a fibrous biomaterial with an anisotropic nature. These two physical quantities have been known to vary significantly with location and sample angle or sample orientation. The tensile strength parallel to the backbone direction can be twice greater than that of the perpendicular direction. The most comprehensive study was reported by Maeser,³ showing that the tensile strength along with modulus and elongation vary in a definitive ellipse pattern with the sample's angle to backbone. This complication creates a great difficulty in fairly representing the fracture resistance of leather based on measurements from one or two sample angles. In a real situation, when a leather sample is subjected to a load, both its stress and strain change simultaneously. Therefore, neither tensile strength nor breaking elongation will really give the whole picture of fracture resistance.

Our ongoing research projects on improving processing and properties of leather have propelled us to look for a physical quantity which can represent better the strength characteristics of leather. This will enable us to more

effectively optimize the leather making processes. The ideal physical quantity should not be sensitive to sampling angles, and should faithfully reflect the fracture resistance of leather. More importantly we hope this physical quantity can be used to characterize the toughness of leather and correlate well with other specific strength requirements, such as tear strength. Consequently, an attempt has been made to develop a new test method for characterizing the fracture resistance. Our approach to this goal is using an energy concept. We have characterized the fracture resistance of leather by measuring the energy needed to fracture a leather sample, which is obtained by integrating the area under the force-elongation curve.⁴ The energy method has been used for other fields such as plastic products, textiles, composites, metals and rubber, and has been proved to be very useful for monitoring the strength quality.^{5,6} In the past, this quantity has been paid little attention by the leather industry. Occasionally only very fragmented data or information was reported. This may be partially due to the enormous time required to calculate the total energy required to fracture the leather, as mentioned before, which is obtained by integrating the area under the force-elongation curve. This has become much more attainable and faster because of recent advancements in microcomputers and software. In theory, fracture resistance characterized by the energy method may be less affected by the anisotropic property of leather because energy is a scalar quantity, whereas tensile stress and elongation are tensor quantities, which are highly direction-dependent. Therefore, an effort has been made to use the energy calculation method to characterize the fracture resistance. In fact, the energy method can also be very useful to characterize the flexibility of leather. By measuring the energy required to stretch a sample to a small strain, one will be able to more readily characterize the resistance of initial deformation, which is strongly associated with flexibility and is more easily performed than using Young's modulus.⁷ This study will be reported in the future. Three major independent variables affecting fracture energy were studied systematically; they are strain rate, moisture content and sample angle. Most of the discussion on the strength characterization in the past has been of a qualitative nature. We have decided to use the technique of experimental design along with factorial analysis to obtain a mathematical model. Therefore, the information can be presented in a quantitative way.⁸ Because of the lengthy calculations involved, the application of those statistical methods seemed to be a cumbersome technique in the past, but the present widespread use of microcomputer and statistical software such as the SAS program has made the task easier, and it is now possible to make calculations in a reliable and rapid manner. We also

studied the effects of fatliquoring and staking on the fracture energy because of their important effects on leather quality. Finally we also present a dimensionless parameter, the ratio of tensile strength to Young's modulus, showing a correlation to the fracture energy. The rationale for this correlation is also elucidated.

EXPERIMENTAL

Materials

A previously frozen mature bovine hide was tanned by the standard ERRC process⁹ being air-dried without fatliquoring and conditioned at 23°C and 50% RH for several months. Rectangular samples having a 10 x 100 mm dimension were cut from the standard test area as described in ASTM D2813-91. These samples without fatliquoring and staking were used in an experimental design as described in the next section. To study the effects of fatliquoring and staking on fracture energy, two sides of mature bovine hide were chrome-tanned by the same method as described before. Each side was then divided evenly between the tail and neck ends, and the tail end of each side was treated to 5% with 2% sulfated Reilly-Whiteman (Conshohocken, PA) fatliquor X-76-31, a "solvent-type oil."¹⁰ Then, one of the sides was dried at constant area at room temperature in still air and then passed through a Molissa staking machine. The other side was dried similarly but without staking, and was used as a control for comparison. These samples were first immersed in a covered culture dish filled with distilled water (approximately 3 times that of the target moisture content by weight) overnight. Samples were then placed in a bench-top vacuum oven to have the moisture content adjusted to desired levels. The moisture content was determined by a leather moisture meter (Delmhorst Instrument Co.) for samples having less than 20% moisture; higher levels were measured by the gravimetric method. The samples were then tested for tensile properties within about 1 minute.

Statistical Design of Experiments

An experimental design from the technique named "response surface methodology," developed by Box and Hunter,¹¹ was applied to the analysis of data. The three factors selected were the strain rate (x_1), moisture content (x_2), and sampling angle (x_3). Originally, there were 23 combinations of factors required by the central composite design. However, we later found that widening the range of moisture content was necessary to obtain a more comprehensive model. Therefore, an additional 16 combinations were

added to the design. Table I is the matrix showing the combinations of coded levels of three factors for each of the thirty-nine experimental conditions. The regression model has the form of a polynomial equation in which the variables are presented as their linear and quadratic terms as well as their bifactorial cross products:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 \quad (1)$$

where Y is the response, such as fracture energy, and b is the individual regression coefficient of the polynomial equation. These coefficients along with analysis of variance can be obtained readily by using the SAS software version 6.11 with a microcomputer. The levels of the coded variables x_1 , x_2 , x_3 , were obtained by means of the following formulae, where x_1' (mm/min), x_2' (%), x_3' (degree, °), are the variables with original scales:

$$\begin{aligned} x_1 &= (x_1' - 200)/100 \\ x_2 &= (x_2' - 15)/3 \\ x_3 &= (x_3' - 0)/45 \end{aligned} \quad (2)$$

The sampling angle is illustrated in Figure 1. The direction perpendicular to the backbone line is designated as 0° , whereas the parallel direction to the backbone line was designated as 90° . The real levels of the variables are listed next to the coded level as shown in Table I.

Measurement of the Fracture Energy

The fracture energy is defined as the energy needed to break the leather. If we consider a leather sample subjected to an extensional force F , increasing in length by a small elongation, dl , we have:

$$\text{energy needed} = F \cdot dl$$

Hence, total energy needed to break the leather (fracture energy)

$$= \int_0^{\text{breaking}} F \cdot dl \quad (3)$$

Which is the area under the force-elongation curve (Figure 2). The force-elongation curve can be readily converted to the so called "stress-strain curve" by simply dividing force by the cross-section area of the test sample for the former, and dividing elongation by the original length of the test samples for the latter. If the thickness and original length of the samples are relatively constant as in this study, then these curves essentially have the same pattern. Therefore, the term "stress-strain curve" may be exchangeable with "force-elongation curve" for describing tensile behavior. We use the term, force-elongation curve, because it is easier to relate to the energy data which is $F \cdot dl$. Other things being equal, the fracture energy of leather will be proportional to its cross-section area (because of the effect on the force needed) and to its length (because of the effect on the elongation). Therefore, the fracture energy is dependent on volume and, consequently, the mass of the tested samples. To compare different samples, we divided the value of Equation 3 by the original mass of the tested area, which is between the clamps of the Instron tester, to present the fracture energy with the SI unit of J/g.

An Instron (model 1122) tensile testing machine was used throughout this work. The energy to fracture along with tensile strength, and breaking elongation were measured as shown in Figure 2. Young's modulus was estimated by measuring the slope of a tangent line for the stress-strain curves from the origin to 10% strain. All the data was calculated and collected through Instron series IX automated materials testing system version 5. The tensile strength of leather was measured at 23°C and 50% RH with a gauge length of 50 mm. The strain rate was adjusted according to the experimental design listed in Table I. The morphology of the fractured ends of the fibers tested at different strain rates was studied using a scanning electron microscope.

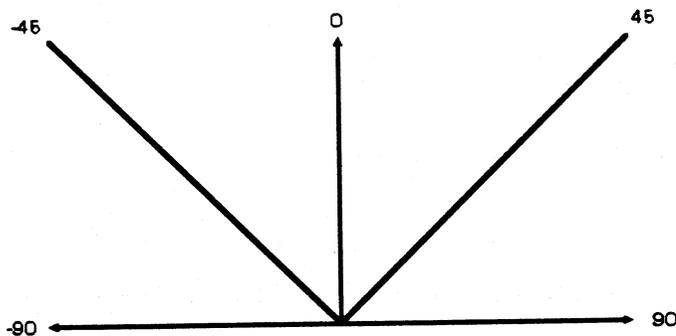


FIGURE 1. — Sampling angle.

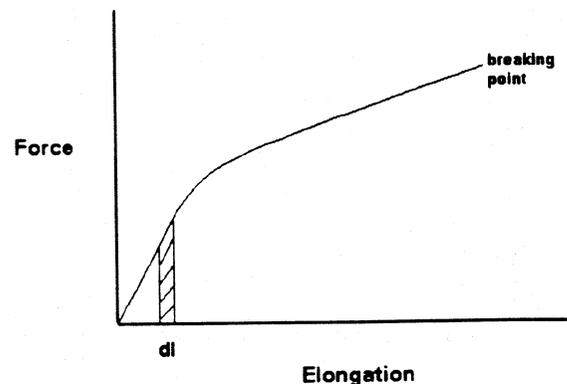


FIGURE 2. — Fracture energy.

TABLE I
Experimental Plan

Run	Coded Values			Strain Rate (mm/min)	Moisture Content (%)	Sampling Angle (°)
	X ₁	X ₂	X ₃	X' ₁	X' ₂	X' ₃
1	-1	1	-1	100	18	-45
2	1	-1	-1	300	12	-45
3	0	1.68	0	200	20	0
4	0	0	0	200	15	0
5	-1	1	1	100	18	45
6	0	0	0	200	15	0
7	0	0	0	200	15	0
8	0	-1.68	0	200	10	0
9	1	1	-1	300	18	-45
10	1.68	0	0	368	15	0
11	0	0	0	200	15	0
12	0	0	1.68	200	15	76
13	0	0	0	200	15	0
14	0	0	0	200	15	0
15	0	0	-1.68	200	15	-76
16	-1	-1	1	100	12	45
17	0	0	0	200	15	0
18	0	0	0	200	15	0
19	0	0	0	200	15	0
20	-1.5	0	0	50	15	0
21	1	1	1	300	18	45
22	-1	-1	-1	100	12	-45
23	1	-1	1	300	12	45
24	0	-0.67	2	200	13	90
25	0	18.33	-2	200	70	-90
26	0	23.67	2	200	86	90
27	0	22.67	-2	200	83	-90
28	0	27	2	200	96	90
29	0	30.33	-2	200	106	-90
30	0	-0.67	-2	200	13	-90
31	0	1.67	2	200	20	90
32	0	4	-2	200	27	-90
33	0	2	2	200	21	90
34	0	10	-2	200	45	-90
35	0	12.67	0	200	53	0
36	0	-5	0	200	0	0
37	0	-5	0	200	0	0
38	0	-5	2	200	0	90
39	0	-5	-2	200	0	-90

RESULTS AND DISCUSSION

Statistical Model and Analysis of Variance

Table II shows the matrix of test results corresponding to the experimental design matrix listed in Table I. The data was processed into the SAS statistical program based on Box and Hunter's design using a microcomputer. Table III shows the regression coefficients of the statistical model, corresponding *t* (Student's *t* values),¹² and significant levels for each coefficient. As indicated in Table IV for the analysis of variance, both the linear and quadratic terms give significant mean square values. The mean square for lack of fit is about the same size as that of pure error, and R^2 (square of correlation coefficient) is 0.76. It is evident that the quadratic model as shown in Equation 4 fits the data fairly well.

$$Y \text{ (J/g)} = 7.51 + 0.55x_1 + 0.39x_2 - 0.18x_3 + 1.89x_1^2 - 0.01x_2^2 + 0.15x_3^2 + 0.12x_1x_2 + 0.20x_1x_3 - 0.013x_2x_3 \quad (4)$$

However, Table IV also indicates that the cross-product term may be neglected because of a high probability of coming from experimental error. A simplified second-order model thus can be expressed as:

$$Y \text{ (J/g)} = 7.51 + 0.39x_2 + 1.89x_1^2 - 0.01x_2^2 \quad (5)$$

Strain Rate

It is well known that the rate of stretching (strain rate) has profound effects on the results of mechanical properties testing. However, the current ASTM test procedure only requires one strain rate, which is 254 mm/min. The effect of strain rate on the mechanical properties of leather has not been subjected to any serious study. There have been some reports describing the effects of strain rate on the fracture behavior of collagen fibers, but there has not been any report on the effect of strain rate on fracture behavior of leather as a whole. Morgan¹³ reported that for untanned collagen fibers taken from cow hide, tensile strength and elongation increase with rate of loading at the beginning, then decrease later when samples were tested at 0, 33, and 66% relative humidity (RH), whereas for samples tested at 100% RH, tensile strength and elongation increased monotonically with strain rate. More recently, Arumugam¹⁴ et al. reported that the tensile strength increased with increasing strain rate when tested at 65% RH for both tanned and untanned collagen fibers taken from the tails of male albino rats. They also described that for chrome tanned fibers, the fracture is closer to a smooth fracture at higher strain rates due to formation of a cohesive unit at the fibrillar end.

Formaldehyde tanned fibers also showed a similar trend. The melting like appearance is also observed at high strain rate. Figures 3 and 4 are the 3-D plots of the response surface according to Equation 4 for 0° and 90° sampling angles, respectively. For both cases, they clearly show that the fracture energy decreases with strain rate at the beginning, then when reaching around 200 mm/min the fracture energy increases with strain rate. This behavior may be explained by the effects of stress concentration and generation of local heat.

At the strain rate between 50 mm/min and 200 mm/min, the effect of stress concentration could play an important role in fracture mechanism. As strain rate increases, the fiber bundles in the leather probably do not have sufficient time to slip and adjust themselves in the stretching direction, and the corresponding stress does not uniformly distribute across the test samples. Stress concentrates at certain regions and consequently causes premature fracture. This behavior can be seen in micrographs of fractured samples. For the 100 mm/min sample shown in Figure 5, the fiber bundles break rather uniformly, while the 200 mm/min sample shown in Figure 6 fractures into finer fiber bundles due to nonuniform stress distribution. Further evidence can be seen in Figures 7 and 8, where the 100 mm/min sample shows a uniform and integral fractured end, while in the 200 mm/min shows the fractured end breaking into separated fibrils. At a strain rate above 200 mm/min, however, the increased friction between fiber bundles may generate sufficient local heat to soften the surface of fiber bundles and make the fiber bundles slip among each other easily, consequently they are able to adjust themselves into better load bearing positions, thus increasing the fracture energy. As shown in the micrograph in Figure 9, the sample was stretched at 368 mm/min, some fibrils seemingly melted on the surface of the fiber bundles. Figure 10 demonstrates the melted-like end of a fractured fiber bundle with broken fibrils fused and twisted together.

Moisture Content

Figure 11 clearly shows that the fracture energy increases with the moisture content of leather. The behavior of water functioning as a lubricant is well known. Water eases the movement of fibers and decreases the frictional resistance of fibers when leather is subjected to a force. The reduction of friction leads to a more uniform stress distribution of stretched leather. Therefore, fracture energy increases with moisture content.

Moreover, the statistical model also indicates that when the moisture content reaches above 98%, the fracture energy

TABLE II
Results

Run	Tensile Strength (MPa)	Break Elongation (%)	Young's Modulus (MPa)	Fracture Energy (J/g)
1	24.1	50.8	51.6	9.81
2	26.9	56.1	60.9	12.20
3	14.3	77.8	14.6	2.00
4	14.0	77.3	21.6	8.27
5	19.0	53.5	38.5	7.44
6	16.5	45.2	40.6	6.14
7	14.6	80.9	20.8	9.58
8	18.2	46.8	50.4	7.05
9	19.2	58.0	31.6	9.02
10	19.4	85.0	30.2	13.72
11	14.0	63.1	28.7	6.54
12	15.5	40.6	44.9	5.14
13	20.8	57.6	41.2	9.78
14	14.1	67.8	23.3	7.64
15	21.7	43.7	60.2	9.25
16	19.0	64.2	35.7	8.86
17	14.6	68.8	34.7	8.27
18	14.0	64.6	24.7	7.68
19	12.3	52.7	36.7	5.30
20	21.1	61.0	28.6	10.84
21	16.8	77.6	23.3	10.89
22	23.4	48.5	68.2	10.03
23	15.6	68.2	31.7	8.36
24	22.0	62.1	46.9	10.95
25	30.0	73.5	26.1	14.65
26	22.0	70.0	22.4	10.14
27	29.8	73.3	22.7	14.62
28	25.0	82.7	16.2	13.94
29	27.6	75.0	22.2	13.35
30	18.7	51.6	45.4	7.79
31	21.2	49.2	40.4	8.24
32	22.6	50.3	40.4	8.89
33	19.8	43.7	55.3	7.30
34	23.4	56.9	43.6	11.12
35	20.5	80.0	20.7	11.92
36	10.9	39.0	57.6	4.16
37	10.9	40.6	57.3	4.49
38	18.3	41.6	53.5	6.61
39	17.7	35.7	64.0	5.75

TABLE III
Regression Coefficients

Parameter	Degrees of Freedom	Regression Coefficient	Standard Error	t for H_0^* : Regression Coefficient = 0	Probability > t**
Intercept	1	7.506	0.40	18.70	0.00
X ₁	1	0.550	0.43	1.29	0.21
X ₂	1	0.393	0.09	4.54	0.00
X ₃	1	-0.182	0.23	-0.79	0.43
X ₁ ²	1	1.887	0.40	4.73	0.00
X ₁ * X ₂	1	0.124	0.54	0.23	0.82
X ₂ ²	1	-0.008	0.00	-2.15	0.04
X ₁ * X ₃	1	0.196	0.54	0.36	0.72
X ₂ * X ₃	1	-0.013	0.02	-0.81	0.43
X ₃ ²	1	0.148	0.17	0.89	0.38

* H_0 = null hypothesis

** t = Student's t = (regression coefficient/standard error)

TABLE IV
Analysis of Variance for Second-Order Model

Regression	Degrees of Freedom	Sum of Squares	R ²	F-Ratio*	Probability > F
Linear	3	149.865		21.149	0.0000
Quadratic	3	66.837		9.432	0.0002
Crossproduct	3	1.971		0.278	0.8407
Total Regress	9	218.674	0.762	10.286	0.0000
Residual	Degrees of Freedom	Sum of Squares	Mean Square	F - Ratio*	Probability > F
Lack of Fit	20	50.385	2.518	1.250	0.3793
Pure Error	9	18.136	2.015		
Total Error	29	68.501	2.362		

* F-Ratio = variance ratio

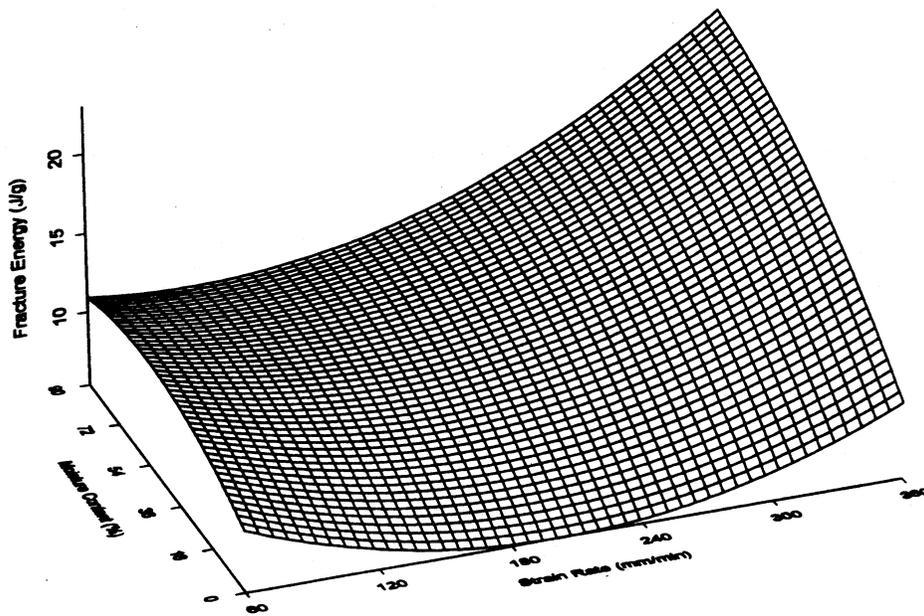


FIGURE 3. — Effect of strain rate for sampling angle of 0°.

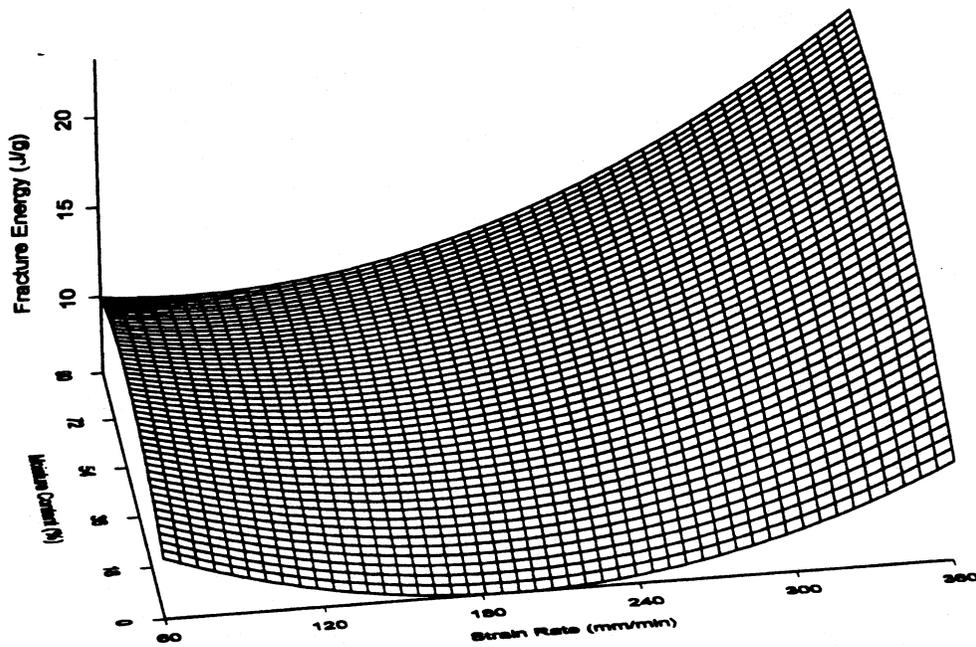


FIGURE 4. — Effect of strain rate for sampling angle of 90°.

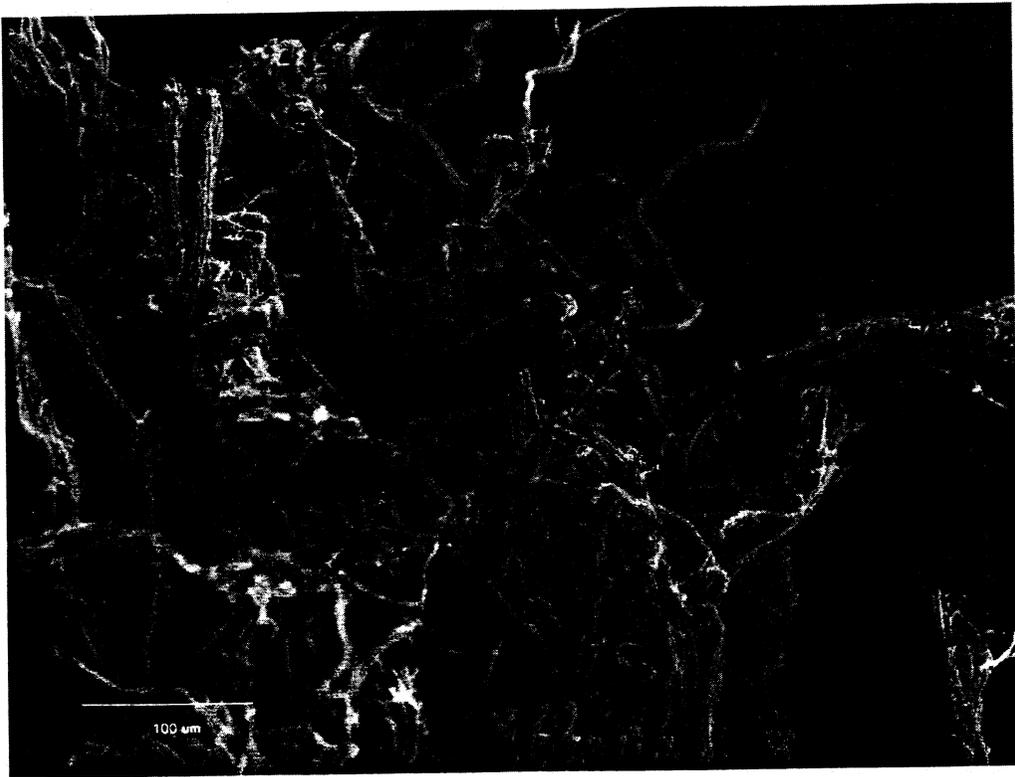


FIGURE 5. — Micrograph of fiber bundles fractured at 100 mm/min.

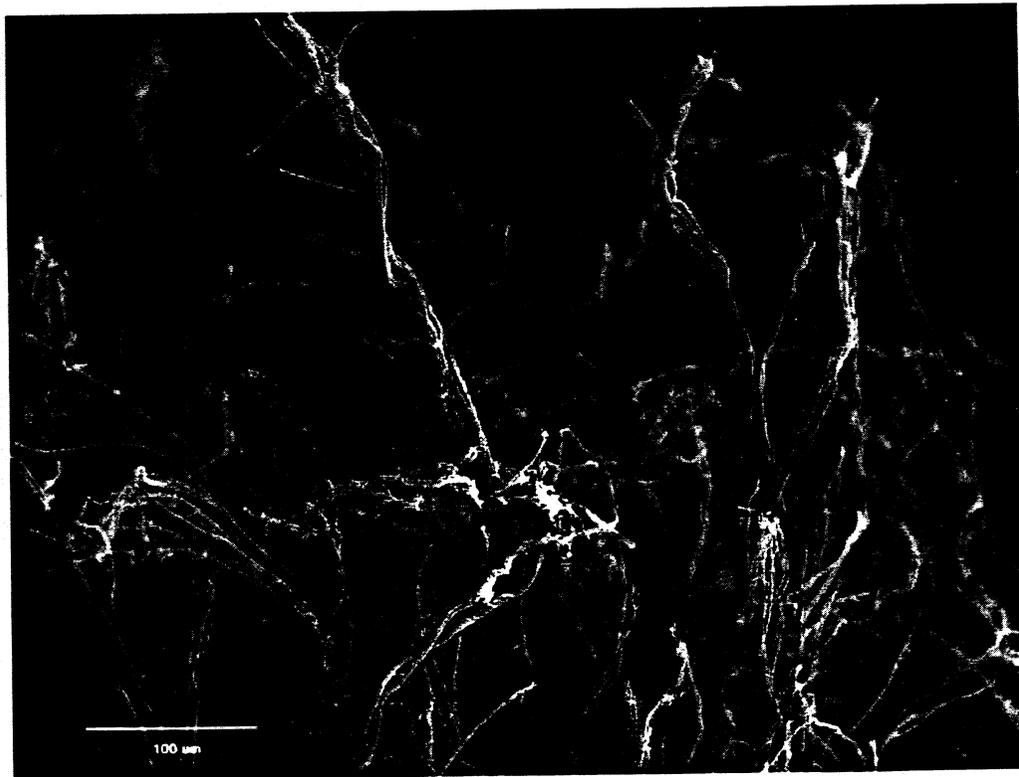


FIGURE 6. — Micrograph of fiber bundles fractured at 200 mm/min.

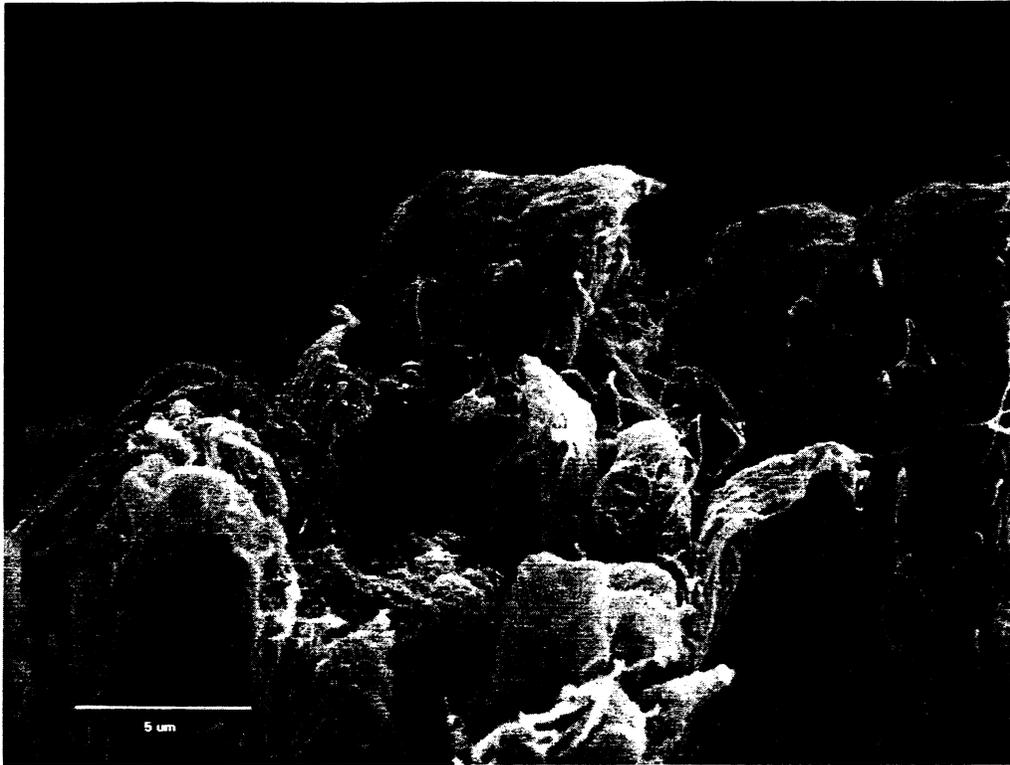


FIGURE 7. — Micrograph of breaking end of a fiber bundle fractured at 100 mm/min.

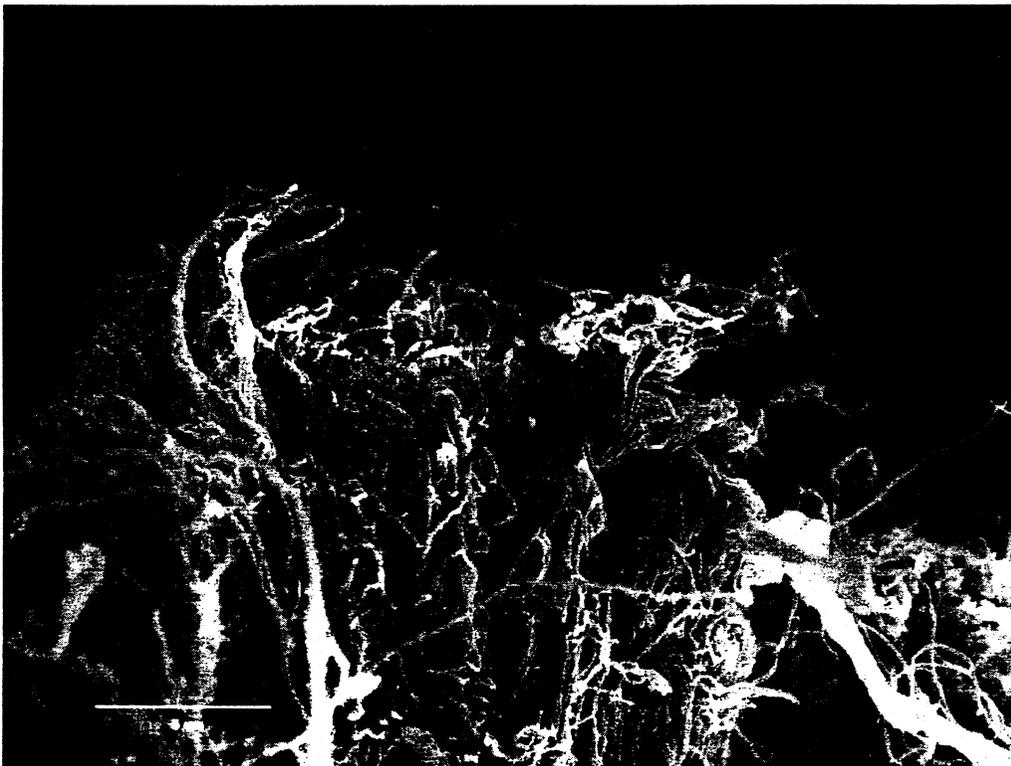


FIGURE 8. — Micrograph of breaking end of a fiber bundle fractured at 200 mm/min.



FIGURE 9. — Micrograph showing melted surface of fiber bundles.



FIGURE 10. — Micrograph showing melted ends of fractured fiber bundles.

starts to decrease. This effect again can be detected clearly in Figure 11. It shows the fracture energy steadily increasing with increasing moisture content then becoming flat at about 90%. Obviously, when leather is over-saturated with water, its fibrous structure becomes loosened and weak.

Sampling Angle

Figure 12 illustrates the fracture energy as a function of sampling angle and moisture content. The effect of moisture content has been discussed previously, whereas here, the sampling angle shows very little effect on fracture resistance. The statistical analysis as mentioned before has indicated that the sampling angle is not a significant factor in determining the fracture energy. Leather is known as a highly anisotropic material. In a very comprehensive study, Maeser has demonstrated that bovine hides have a highly anisotropic structure and their tensile strengths are very sensitive to the sampling angle.³ This is not surprising, because tensile strength is a vector quantity. It only quantifies breaking stress in the one direction that it is being stretched. Therefore, the resistance is given only by the vector components parallel to the applied force direction.

Obviously, the more fiber bundles that line up in this direction, the higher the tensile strength that is attained. Our tests have also shown that the tensile strength is greater in the parallel direction than the other sampling angles. This can be seen clearly in Figure 13, where the sample at the angle of 0° is the weakest and those at 90° or -90° have the highest tensile strength. In fact, there is an almost perfect symmetric pattern with a minimum tensile strength at 0° . This agrees with Maeser's early finding. However, this is not true for fracture energy which seems almost independent of sampling angle.

Toughness Index: A Dimensionless Parameter

This study has demonstrated that leather strong in terms of tensile strength is not necessarily strong in terms of fracture energy. For example, a brittle leather may have excellent tensile strength, but it may have poor fracture energy. As mentioned before, the fracture energy is a measure of the integral of the force-elongation curve. There are two physical quantities that combine to produce energy, i.e. force and elongation. Fracture energy counts the summation of energy required for every fiber bundle movement until it

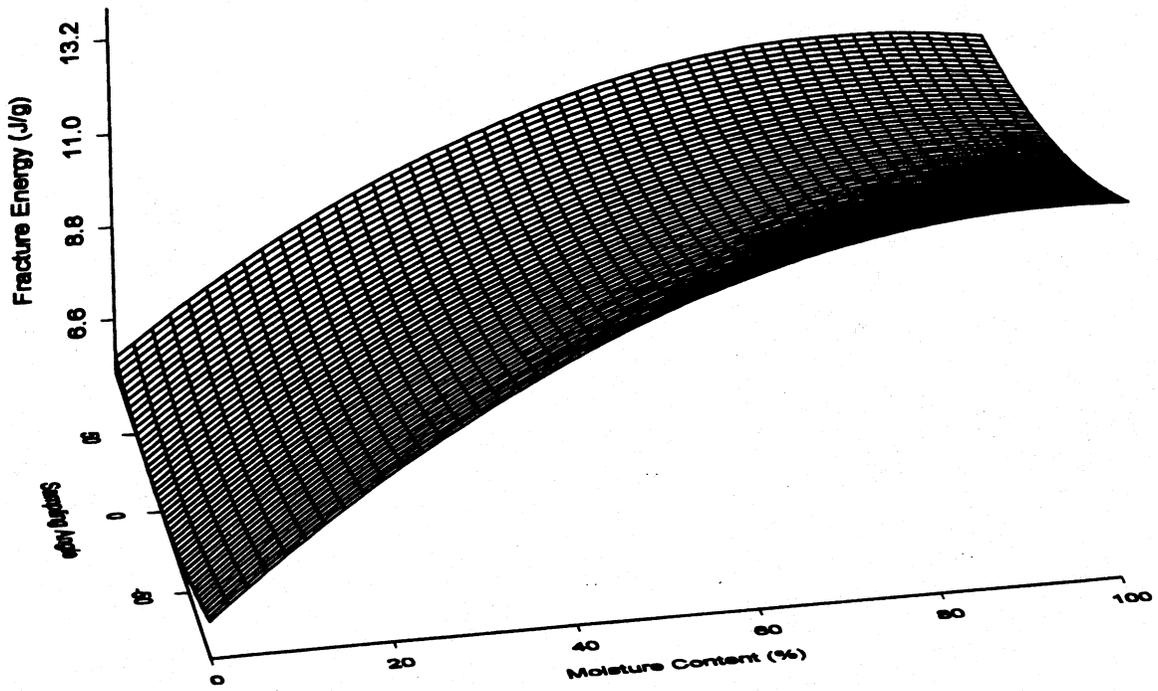


FIGURE 11. — Effect of moisture content on fracture energy.

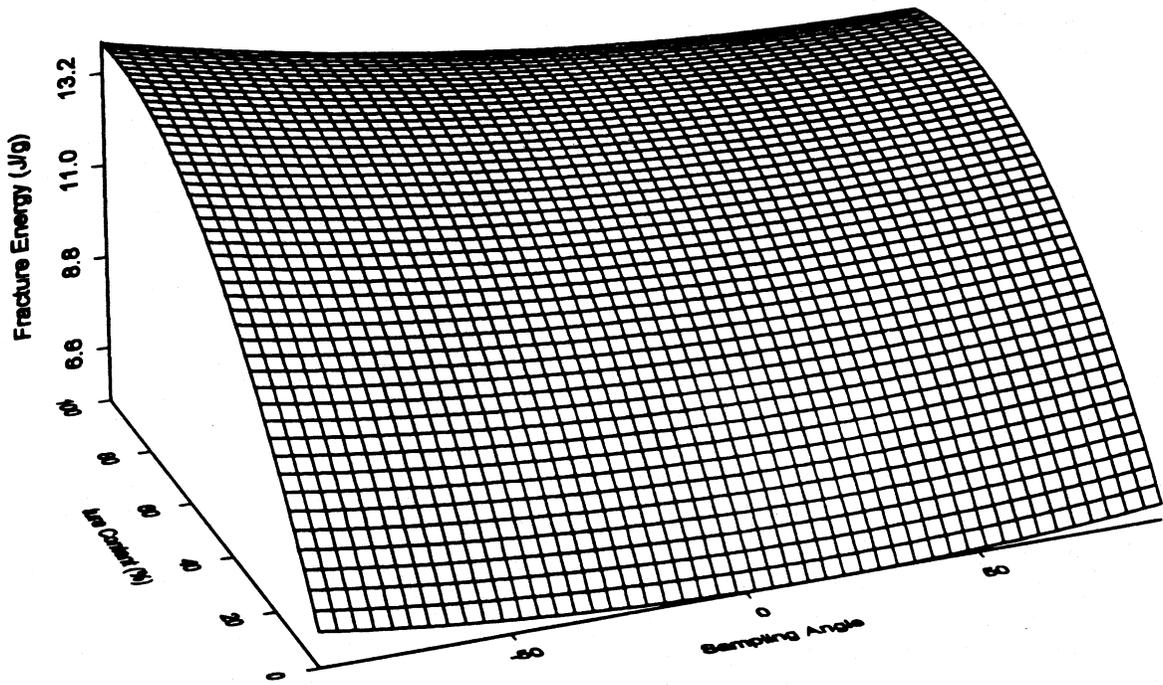


FIGURE 12. — Effect of sampling angle on fracture energy.

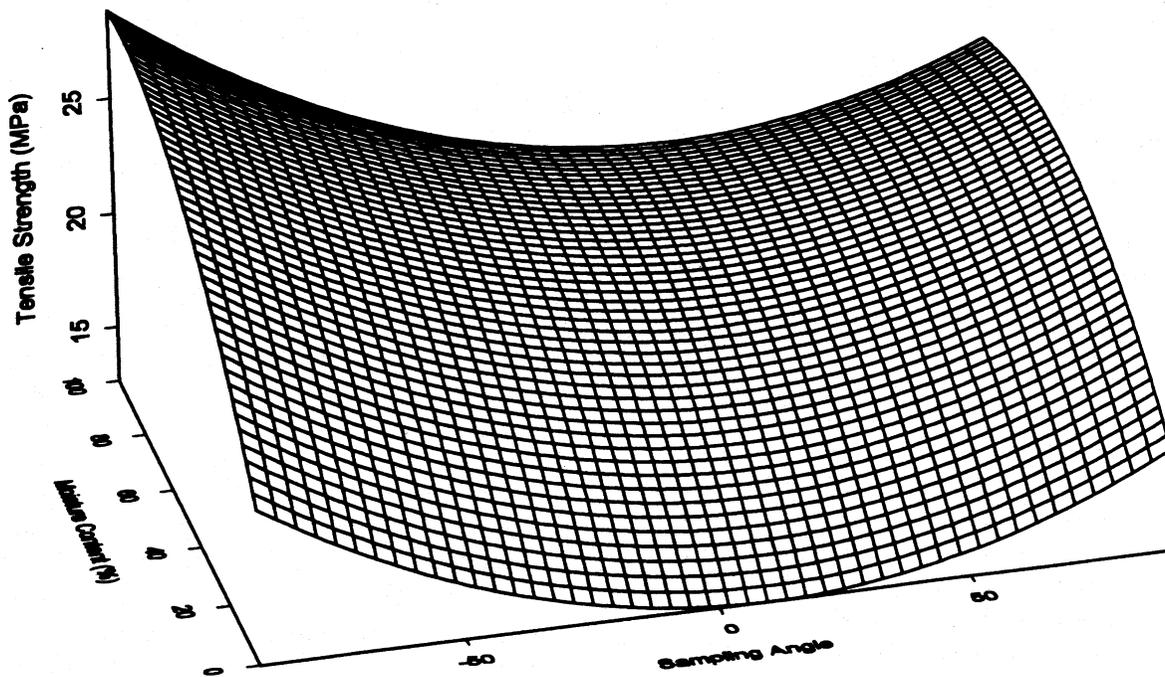


FIGURE 13. — Effect of sampling angle on tensile strength.

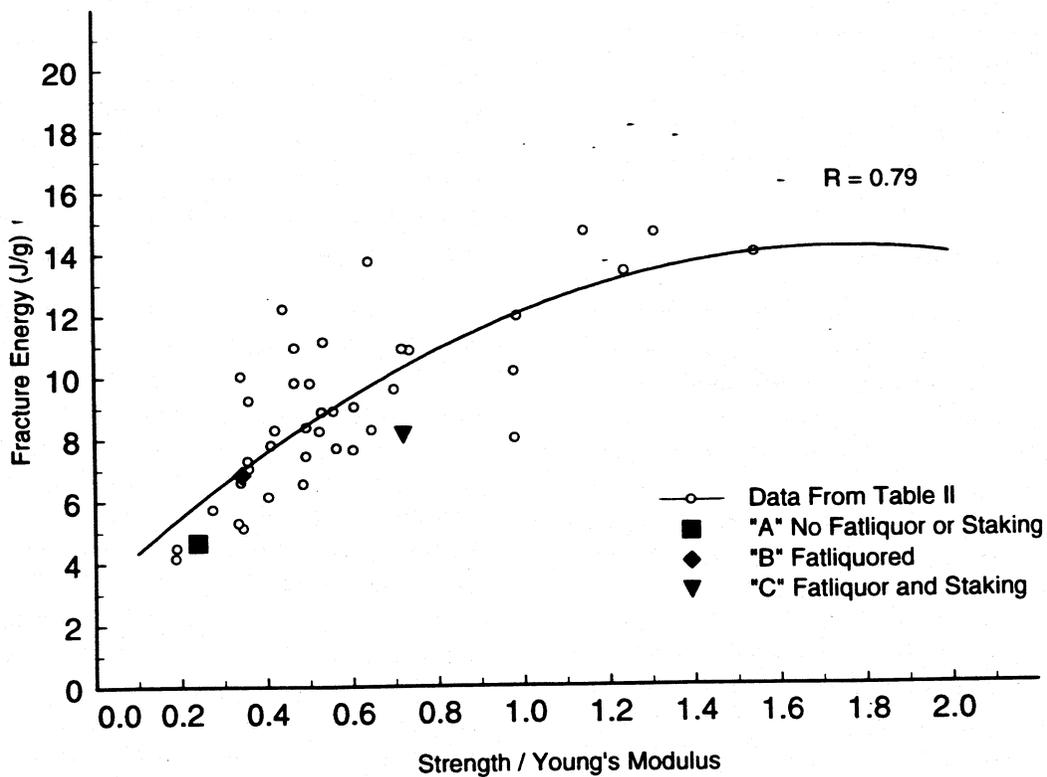


FIGURE 14. — Fracture energy vs. the ratio of tensile strength to Young's modulus.

fractures. Therefore, force and elongation (or stress and strain) are taken into account together from beginning to end. This is in contrast to tensile strength, which is only a measure of the stress required to fracture a material. Leather products, in general, require not only good tensile strength but also flexibility, especially for the garment and automotive industries. Both strength and compliance are product criteria. In other words, the tensile strength and compliance should be considered together. We may put these two factors together as one factor by using their cross product, i.e. strength x compliance. The compliance can be best represented by the reciprocal of Young's modulus. Thus, this cross product becomes the ratio of tensile strength to the Young's modulus. If a material is strong and yet not brittle, we may say that it is tough. Therefore, we name this ratio as the toughness index because this ratio gives quantitatively the degree of toughness. It is interesting to note that this ratio is dimensionless. It is independent of the geometry of the leather samples. Therefore, even without knowing the thickness or shape of the samples, one can still make an effective comparison of properties. Dimensionless parameters have been showing their importance in various characterizations of materials or processing, such as ratio of length to diameter of fibers, Reynolds number, etc. Figure 14 illustrates a correlation between fracture energy and toughness index. It shows a general trend that the higher the toughness index the greater the fracture energy. It is clear that the fracture energy is associated with the toughness of leather. In fact, in some materials science literature the terms fracture energy and toughness are synonymous.^{15,16,4}

Effects of Fatliquor and Staking

Fatliquoring has been practiced in leather making to improve the flexibility and softness of leather. It is an oil adding process by which the leather fibers are lubricated so that after drying they will be capable of slipping over one another. Its pronounced effect on leather properties can be seen clearly in Figure 14, where A is the control sample, and B a sample that has been fatliquored as described in the experimental section. Both toughness index and fracture energy are much greater than those of the control.

Staking is an additional process in leather making to enhance the pliability of leather. In combination with the correct fatliquoring treatment, staking governs the final firmness or softness of the leather. In staking, leather is subjected to a very large number of rapid oscillations, stretching and flexing in every direction. This mechanical

action is necessary to break weak adhesions within the fiber structure, thereby promoting fiber mobility.¹⁷ The mechanical stresses that staking imposes on the leather are very great, and if it is overdone it can adversely affect leather integrity.¹⁸ As shown in Figure 14, sample C has been subjected to staking with fatliquoring and yields increased fracture energy and toughness.

CONCLUSIONS

Using the energy approach to characterize the fracture resistance is well established in materials science.^{15,16,4} It is particularly useful to deal with anisotropic materials such as skin, hides and leather. In the past, however, very little attention has been given to this physical quantity by the leather industry. In this paper we have utilized the technique of experimental design and statistical analysis to mathematically model the influence of strain rate, moisture content and sampling angle upon the fracture energy. Results show that strain rate is a complex factor acting upon fibrous materials such as leather. The fracture energy at first decreases then increases with increasing strain rate. The increasing heat generated during high speed stretching induced an effect of plastication upon the fiber bundles, therefore increasing the fracture resistance of leather. Water acts as a plasticizer lubricating the fiber bundles and enhancing the resistance to fracture, however when it reaches a certain limit, it has an adverse effect on leather integrity. Most importantly, contrary to tensile strength and breaking elongation, the sampling angle has shown little effect on the fracture energy. Staked and fatliquored leather clearly showed improved fracture energy in our study.

In this report we have also described a dimensionless parameter, the ratio of the tensile strength to Young's modulus. It provides a quantitative expression of the toughness of leather, therefore we named it the toughness index. A correlation has been demonstrated between this index and fracture energy. Actually, the term fracture energy has been used interchangeably with "toughness." It is obvious that toughness is an important criteria in most leather products such as upholstery and garments. In other words, fracture energy is the leather property that one really needs to be concerned about, not the tensile strength or breaking elongation alone. It appears that good fracture energy really reflects a superior balance of strength and flexibility. We hope to draw the leather industry's attention to the importance of fracture energy in regards to the leather it manufactures.

ACKNOWLEDGMENTS

We wish to thank Dr. John G. Phillips for advice on statistical experimental design and the use of the SAS program. We also thank Dr. Peter H. Cooke and Mr. Lenier W. Tucker, Sr. for the SEM micrographs. Particular appreciation is extended to Drs. Jifeng Ding and Geoff E. Attenburrow at British School of Leather Technology for their invaluable suggestions.

REFERENCES

1. American Society for Testing Materials, Annual Book of ASTM, Vol 15.04, D2209-95, 1995.
2. American Society for Testing Materials, Annual Book of ASTM, Vol 15.04, D2211-95, 1995.
3. Maeser, M.; "The Effect of Hide Location and Cutting Direction on the Tensile Properties of Upper Leathers," *JALCA* **55**, 501-530, 1960.
4. Morton, W. E., and Hearle, J. W. S.; "Physical Properties of Textile Fibers," The Textile Institute, Manchester and London, **271**, 1978.
5. Treloar, L. R. G., and Riding, G. J.; "A Theory of the Stress-Strain Properties of Continuous-Filament Yarns," *Textile Inst.* **54**, T156, 1963.
6. McCrum, N. G., Buckley, C. P., and Bucknall, C. B.; "Principles of Polymer Engineering," Oxford Univ. Press, New York, 187, 1992.
7. Liu, C. K. and Brewer, J.; *U. S. Patent* 5,217,485, June 8, 1993.
8. Cochran, W. G. and Cox, G. M.; "Experimental Designs," John Wiley & Sons, New York, 335, 1951.
9. Taylor, M. M., Diefendorf, D. J., Hannigan, M. V., Artymyshyn, B., Phillips, J. C., Fairheller, S. H., and Bailey, D. G.; *JALCA* **81**, 43-61, 1986.
10. Kronick, P. L., Page, A., and Komanowsky, M.; "An Acoustic Emission Study of Staking and Fatliquor." *JALCA* **88**, 178-186, 1995.
11. Box, G. E. P., and Hunter, J. S., "Multifactor Experimental Designs," *Ann. Math. Stat.* **28**, 1957.
12. Steel, R. G. D., and Torrie, J. H., "Principles and Procedures of Statistics," McGraw-Hill, New York, 43, 1960.
13. Morgan, F. R., "The Mechanical Properties of Collagen and Leather Fibers," *J. Soc. Leath. Tech. Chem.* **55**, 4-23, 1960.
14. Arumugam, V., Naresh, M. D., Somanathan, N. & Sanjeevi, R., "Effect of Strain Rate on Crosslinked Collagen Fibers," *J. Soc. Leather Technol. Chem.* **79** (5), 143-147, 1995.
15. Van Vlack, L. H., "Elements of Materials Science and Engineering," 4th ed., Addison-Welsey, Reading, Massachusetts, 10, 1980.
16. Stippes, M., Wempne R. G., Stern, M., and Beckett, R.; "The Mechanics of Deformable Bodies," Charles E. Merrill, Columbus, Ohio, 67, 1961.
17. Kronick, P. L. and Page, A.; "Recovery of Properties of Staked Leather on Storage," *JALCA* **91**, 39-46, 1995.
18. Alexander K. T. W., Covington, A. D., and Stosic R. G., "The Production of Soft Leather Part 2. Drying and Stress Softening," *JALCA* **88**, 271-277, 1993.

DISCUSSION

Ann Stanley, BLC: We have been doing some work on improving the peel strength and tear strength using polymers. One of the problems that we have is with our control samples. I noticed that your control samples were unfatliquored and air dried. Now I would highly recommend that you freeze dry the samples because in your air dried samples your fibril bundles will be stuck together and you will actually be artificially altering the strength properties. In order to do the test, you will actually have to physically separate the fibril bundles as in the photographs that I showed earlier. So your control samples will be best placed if you use freeze dried samples because this would eliminate any problem with the fibrils actually sticking and you may find that you will get slightly different results. It would be much more representative of what really happens in an unfatliquored state sample.

That is true, that is very true. In my studies, the only difference between the fatliquored and the control sample is just the fatliquor.

Ann Stanley, BLC: But that is the crucial difference because if you don't have the fatliquor there, unless you freeze dry, those fibril bundles will be stuck together. Because the fat replaces the water, you can eliminate that problem.

Yes, you are right. This study is trying to establish a trend; whether there is any correlation to tear strength; whether there is an effect of sampling angle, any correlation with the

toughness. Those were my objectives for this study. More studies are coming in which I will take your advice.

Sam Schneider, retired: The true test of whether leather is strong or not is made in the shoe factory by a lasting machine. Either it lasts or it doesn't last. There is a large difference in where you take your samples in a side because the fiber structure varies from the belly to the backbone. The belly is so much stronger because of the way the fibers grow whereas in the backbone you may have such weak leather that you can tear it apart with your little finger. So, the test of direction and the test that you make with water and with fatliquor are really insignificant in the practical

making of leather because most tanners know how to make leather fatliquored properly. Nobody is making leather without fatliquor. The fact is that part of our problem in the leather industry, as I see it, is that people do not do things in a practical manner. So everything has to be checked from that point of view as to the end results.

You are right. This study is trying to demonstrate that you can correlate data to the toughness of your leather. That was one of my objectives to establish a trend. I think that certainly if you are talking about location of the sample. The whole study by ASTM is the sample location. You must use a standard test area.