

ACOUSTIC EMISSION STUDIES FOR LEATHER USING DUAL SENSORS

by

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ABSTRACT

Since leather is sold by the square foot, destructive tests lessen the square footage of the material and infringe on the leather manufacturer's total profit. Therefore there is a need for developing an instrument to perform nondestructive testing of the physical properties of leather. In this investigation, experiments were designed to pass leather test samples through a pair of rotational acoustic sensors, thereby enabling the collection of acoustic emission (AE) signals from both the grain and corium and provide a more accurate assessment of the quality of leather. Observations showed a strong correlation between the mechanical properties of leather and the corresponding cumulative acoustic energy. We also used this dynamic method to characterize the grain break of leather. Results showed that the difference in grain break could be determined from the amount of acoustic hits collected from moving the AE sensors over a leather sample. Observations showed the poorer the grain break; the more AE energy was detected. Data also demonstrated that thicker samples tended to have poorer grain break. In short, this study demonstrated that the tensile strength, stiffness, toughness and grain break could be nondestructively determined by measuring acoustic quantities with a pair of rotational sensors.

RESUMEN

Dado que el cuero es vendido por el pie cuadrado, los ensayos destructivos disminuyen el pietaje del material y atentan contra la ganancia total del fabricante del cuero. Por lo tanto hay una necesidad de desarrollar un instrumento para llevar a cabo ensayos no destructivos de las propiedades físicas de cuero. En esta investigación, los experimentos fueron diseñados para pasar la prueba de muestras de cuero a través de un par de sensores acústicos rotativos, lo que permite la recolección de señales de emisiones acústica (EA) tanto de la flor como del corion y proporcionan una evaluación más precisa de la calidad del cuero. Las observaciones mostraron una fuerte correlación entre las propiedades mecánicas del cuero y la correspondiente energía acústica acumulada. También hemos usado este método dinámico para caracterizar el quiebre de la flor de cuero. Los resultados mostraron que la diferencia de quiebre de la flor podría ser determinada por la cantidad de impactos acústicos recogidos moviendo los sensores de EA sobre una muestra de cuero. Las observaciones mostraron que cuanto más pobre es el quiebre del cuero mayor energía EA fue detectada. Los datos también demostraron que las muestras más gruesas tienden a tener peor quiebre de flor. En resumen, ese estudio demostró que la resistencia a la tracción, la rigidez, la resistencia y el quiebre de la flor podría ser determinado por ensayos no destructivos midiendo las cantidades acústicas con un par de sensores rotativos.

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INTRODUCTION

As with any other manufactured product, leather must meet certain quality criteria. Quality control and assurance procedures in the leather industry today require destructive tests on finished leather in order to determine its material properties. These tests are performed prior to leather's being made into a final product. Since leather is sold by the square foot, the destructive tests lessen the square footage of the material and therefore infringe on the leather manufacturer's total profit. Therefore there is a need for developing an instrument to perform nondestructive testing of the physical properties of leather. We are presenting a method that may potentially be a real-time (during the production process) nondestructive Testing (NDT) method for evaluating the material properties of leather as a quality control procedure, while avoiding damage and waste of materials.

Acoustic Emission (AE), as a method of nondestructive testing, has been used for many years in a wide variety of applications including inspection of aircraft and military equipment, civil infrastructure, pipelines and tubes, petrochemical facilities, and industrial systems. We have recognized acoustic emission as a useful method for characterizing leather properties.^{1,2} An initial feasibility study into the use of AE for leather characterization established that AE is a promising technique for the assessment of leather properties.³ The potential of using AE as a quality assurance (QA) and quality control method led us to research some of the problems related to the different stages of the leather making process. Some interesting results have been obtained from the use of AE for studying the degree of opening-up of the leather structure obtained by the liming process.⁴ Also, AE has been applied in the study of leather tear resistance, tensile strength, degree of lubrication, and quality of final coatings.^{5,6} Most recently, we have advanced further in our AE investigations using a nontraditional sensor, a rotational sensor.⁷ Our objective for this research project is the production of an AE tester that can nondestructively measure the quality of leather. We examined the feasibility of using the AE technique to nondestructively measure the mechanical properties of leather, particularly tear strength and softness. Both properties are critical for leather, especially for upholstery and automobile applications. We used a rotational acoustic sensor that contacted the leather samples to collect their AE quantities and properties.⁷ Our previous observations showed an excellent correlation between the tear strength of leather and the corresponding acoustic counts, read from an AE analyzer. Data also indicated a close relationship between the softness of leather and the corresponding ratio of acoustic hits to energy. The observed phenomena indicated that the tear strength and softness of leather could be nondestructively determined by measuring the acoustic quantities with a

rotational sensor crossing over the leather to be tested. We also extended AE studies to characterize the fracture mechanisms of various green composites (bio-based and bio-degradable).⁸⁻¹¹

In this paper we will present recent AE research using a pair of rotational AE sensors to characterize the leather properties. The leather samples traveled between the sensors, thereby enabling the collection of AE signals from both sides of the leather samples. We hope by this unique setup, one will be able to more accurately measure the leather properties.

EXPERIMENTAL

Materials

Finished chrome-tanned bovine leathers were used for this investigation. The samples' thickness ranged from 1.0 to 1.2 mm, which is typical for upholstery and automobile applications. Before physical property testing, the samples were stored in a conditioned room at 23°C and 50 % RH according to ASTM standard method D1610-96, "Standard Practice for Conditioning Leather and Leather Products for Testing." The moisture content of samples was determined to be 15 ± 1 % by a moisture meter for leather (Delmhorst Instrument Co., Towaco, NJ).

AE Methods and Apparatus

Leather is a fibrous collagen material. Sound waves are longitudinal mechanical waves. Transient sound waves can be generated in the region of a material that experiences abrupt changes in strain. This phenomenon is known in materials science as acoustic emission (AE) and is generally detected by means of ultrasonic transducers coupled to the material. Typical examples of events which produce acoustic emission are fiber movements and breakages, interfacial bond failure in fiber composite materials, growth of microcracks, and delamination of thin films. This basic phenomenon may be defined as an AE event, which is translated by an AE analyzer as a "hit".¹² The operating frequencies of sensors used in AE measurements are usually in the range of 50 - 1000 kHz, which is well above the audible sound frequency. This permits all ambient noise to be filtered out, leaving only the frequencies of interest. When the AE sensor detects a signal over a certain level (i.e., the threshold), an AE event is captured. The amplitude of the event is defined at the signal peak. A single acoustic emission event (hit) may consist of many emission counts, which are the number of times a signal from the transducer crosses a preset amplitude threshold, as illustrated in our previous report.¹ The AE sensors are generally very sensitive piezoelectric sensors. Since the AE signals are often weak, a preamplifier is connected right after the AE transducer to minimize noise interference and prevent signal loss. The main amplifier amplifies the signals before being sent to the signal analyzer. After that, the AE quantities are stored in a

computer for further analysis. Each acoustic hit generates a wavetrain from the transducer consisting of a number of oscillations (waves), so-called "acoustic counts." The hits with high amplitudes always produce high numbers of counts.¹³

A schematic diagram of the experimental setup is shown in Figure 1. This set-up is very different from our previous experiments. Here leather samples travel through a pair of AE sensors and AE signals are captured and analyzed for both the grain and corium. We believe this setup will give a more accurate evaluation because the AE signal contributions are coming from both the grain and corium.

The AE sensor is the single most important factor in AE testing because it defines the operating and resonant frequency range and the sensitivity of the signals collected. In our earliest work, we clipped a small piezoelectric AE sensor against the leather sample to obtain AE data. To perform nondestructive tests, we changed our sensor from a traditional flat static sensor to a rotational sensor. This rotational (rolling) sensor was designed to serve in a dry-couplant application that requires dynamic measurements. The sensor's multi-element construction, coupled with advancements in acoustic impedance matching, provides high sensitivity with low noise. This sensor offers several key advantages in automated and moving process applications. First, the need for a couplant, such as vacuum grease (that enhances transmission of acoustic waves from test samples to sensor) becomes obsolete, because the rolling sensor's compliant tire makes contact with the part being monitored. Second, this sensor maintains a constant distance to a moving sample and, therefore, constant sensitivity. Because the rotational sensor is not fixed to the test surface, it can be applied automatically for each new process. The leather samples were examined using a dual sensor assembly as shown in Figure 1, and each sample was examined over a total scan length of 61 cm (24 inches), 30.5 cm (12 inches) each from different parts of the sample. The sensors were moved along the surface of the

samples with a 10 N force applied to the leather and traveled at the rate of 1.3 cm (0.5 inches) per second, and the hit threshold was set at 35 dB. The acoustic signals (waveforms) were carefully analyzed in order to extract as much information as possible.

AE data were processed with a preamplifier and a LOCAN-AT Model 3140 acoustic emission analyzer (Physical Acoustics Corp.). Each acoustic hit from an acoustic event in the sample causes a damped oscillation to be emitted by the transducer. The analyzer records the duration of each oscillation hit, its amplitude, and its energy (10^{-6} dB.s). Only hits giving maximum amplitudes greater than 35 dB (threshold) from the sensor were counted. All the reported AE quantities in this paper are cumulative values, i.e. they are the total values cumulated from the beginning to the end of the AE tests. To normalize results from various samples, we divided the AE data by the samples thickness. The AE energy (10^{-6} dB.s) of the hits is commonly reported as the average area under the envelope of the waveform, so the hit amplitudes and hit durations determine the energy.¹³

Scanning electron microscopic examinations were conducted on the cross section of leather samples to examine the degree of opening up in the fibrous structure. Samples were mounted on aluminum specimen stubs using colloidal silver adhesive (Electron Microscopy Sciences, Ft. Washington, PA) and sputter-coated with a thin layer of gold. Images were collected using a Model JSM 840A scanning electron microscope (JEOL USA, Peabody, MA), integrated with a model Imix 1 digital image workstation (Princeton Gamma-Tech, Princeton, NJ), and operated in the secondary-electron imaging mode.

Tensile Property Measurements

Besides nondestructive AE tests, we also measured the tensile strength, Young's modulus and fracture energy of the leather samples used in this investigation. Tensile strength is the maximum stress in tension that the leather may sustain without breaking. Young's modulus is a physical quantity representing the stiffness of a material. It is determined by measuring the slope of a line tangent to the initial stress-strain curve. Rectangular shaped leather samples (1- × 10-cm) were cut near the standard test area as described in ASTM D2813-97, "Standard Practice for Sampling Leather for Physical and Chemical Tests," with the long dimension parallel to the backbone. These properties were measured with a sample length of 5 cm between the two grips. The strain rate (crosshead speed) was set at 5 cm/min. An upgraded Instron mechanical property tester, model 1122, and Testworks 4 data acquisition software (MTS Systems Corp., Minneapolis, MN) were used throughout this work. Each test was conducted on five samples to obtain an average value. We used data analysis and graphics software, Axum version 6 (MathSoft, Inc, Cambridge, MA.) to derive regression models and construct 2-D or 3-D regression plots. The regression equations and corresponding correlation coefficients are listed in the plots.

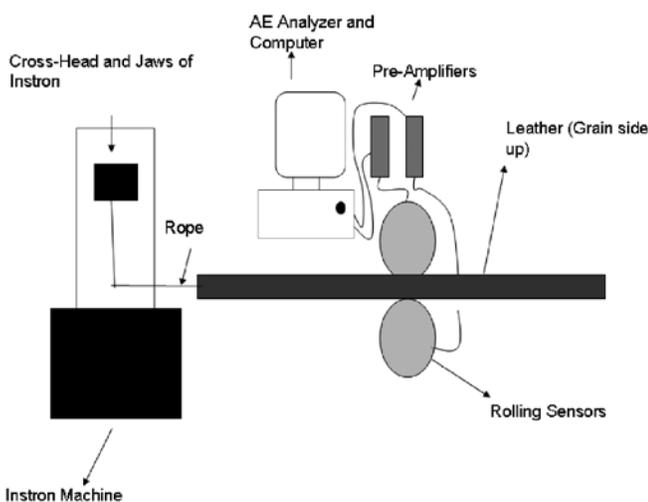


Figure 1. – Nondestructive testing system with dual AE sensors.

RESULTS AND DISCUSSION

As demonstrated in Figure 2, the grain and corium have drastically different textures; the grain (Figure 2a) has a much smoother surface, whereas the corium has a relatively rough surface. The detailed difference between these two layers is shown more clearly in a cross-sectional view as Figure 3. The grain layer shows the fibers forming a sheet-like layer with very little connection in between, whereas the corium shows an intense network structure with fiber bundles interwoven together.

Figure 4 demonstrates a close relationship between the total AE hits and energy obtained from both grain and corium. It implies that the higher energy sound waves will produce more hits. However, as shown in Figure 5, the effect of AE energy on total hits from the corium is much greater than the grain. This is ascribed to the fact that the diameters of fiber bundles are larger in the corium than in the grain. The grain as reported in our previous paper has a finer fibrous structure that may produce less AE energy and hits.⁵



Figure 2. – (a) Grain face and (b) Corium face of leather; photos were taken by using a digital camera.

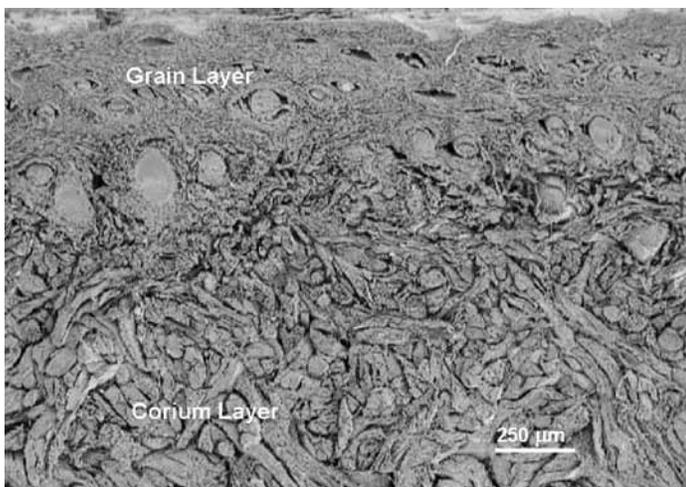


Figure 3. – Cross-sectional view of leather.

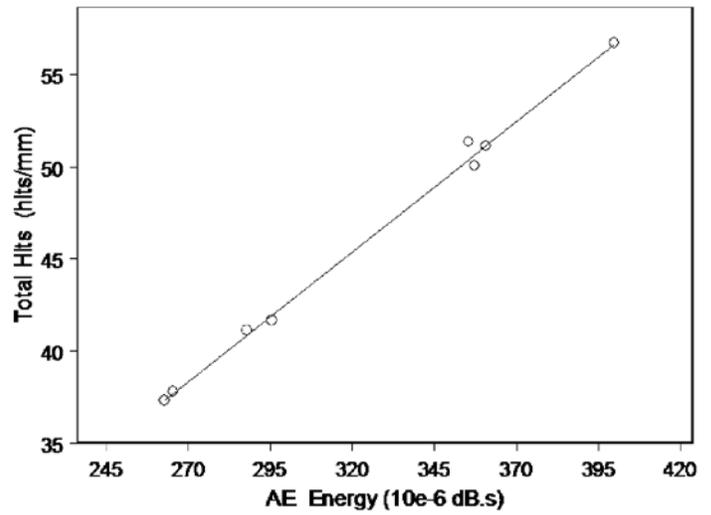


Figure 4. – Total Hits vs. Total Energy.

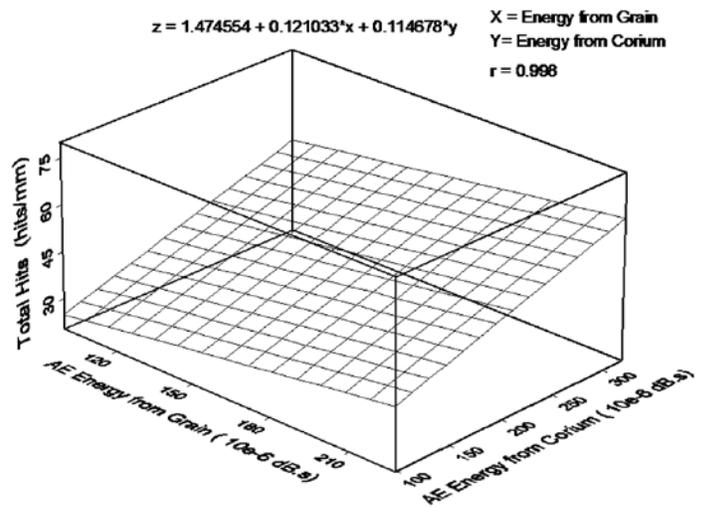


Figure 5. – Total hits as a function of AE energy from grain and corium.

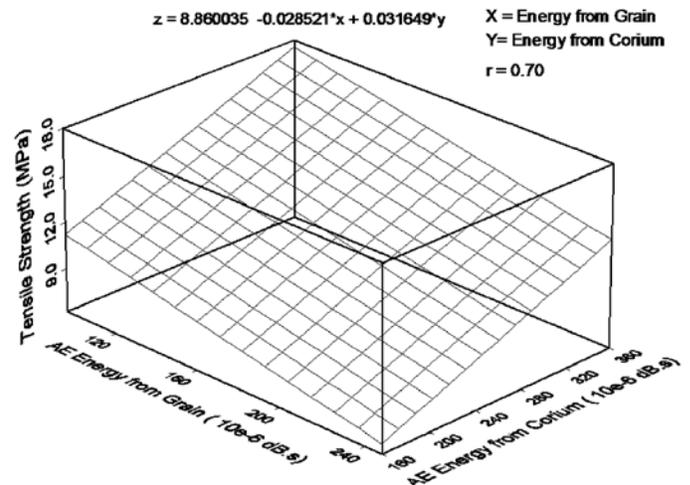


Figure 6. – Tensile strength as a function of AE energy from grain and corium.

Correlation between AE Quantities and Physical Properties

The regression equations of physical properties were derived as a function of various AE quantities such as energy from grain (EG), energy from corium (EC), hits from grain (HG), hits from corium (HC), total counts (TC) and thickness of samples (D) as shown in Table I with the corresponding multiple correlation coefficients (r).

Figure 6 shows a regression plot of tensile strength as a function of AE energy from grain and corium. It shows the tensile strength has a positive correlation with AE energy from the corium, whereas with the grain the tensile strength has a negative correlation. The reason for this difference is ascribed to their structural difference. The greater amount of AE energy reflects a denser fibrous structure, which results in a larger tensile strength. However, this principle doesn't apply to the grain, because the denser grain structure often indicates a rigid fibrous structure in which the fibers are finer and are arranged in a sheet-like structure, which has a negative impact on the tensile strength. Importantly, the results demonstrated in Figure 6 imply that the tensile strength of leather can be predicted by the AE nondestructive test method described here, by measuring the AE energy. Tensile strength determines the maximum tensile stress the leather can sustain without fracture. Adequate tensile strength is very important in manufacturing leather goods, where the leather is often subjected to a tensile force during mechanical stretching or elongation. Moreover, in a variety of end uses, leather goods must be capable of resisting considerable stress without fracture.

Leather Stiffness

Young's modulus is one of the most important physical quantities characterizing the mechanical properties of leather. It expresses the resistance of leather subjected to a small

TABLE I

The multiple correlation coefficients (r)

Physical Property	Multiple Regression Equations	r
Tensile Strength	8.86 - 0.0285 GE + 0.0316 CE	0.70
Young's Modulus	-6.6151 + 0.0093 GH + 0.3031 CH	0.69
Fracture Energy	-4.3452 - 0.1015 GH + 6.6714 D	0.76
Grain Break	11.3564 + 0.0724 GH - 7.828 D	0.71

GE=Grain Energy, GC=Corium Energy, GH=Grain Hits, CH=Corium Hits, D=Thickness

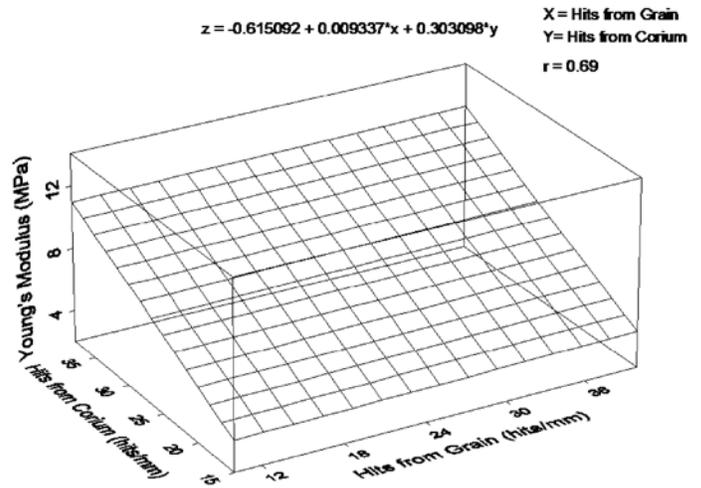


Figure 7. – Young's modulus as a function of AE hits from grain and corium.

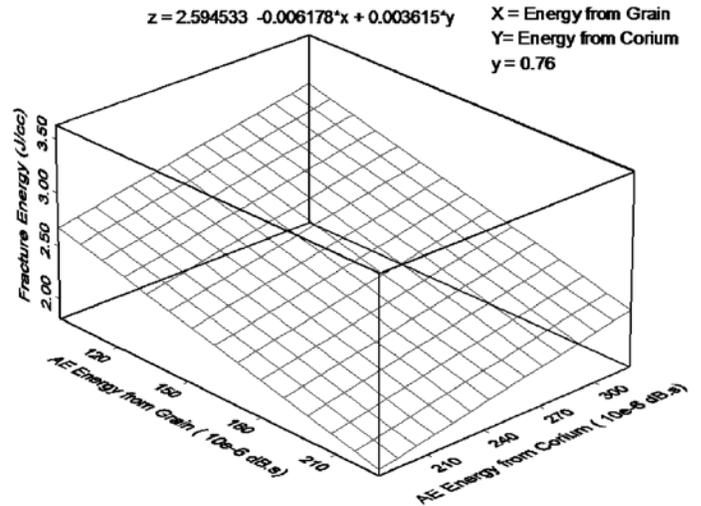


Figure 8. – Fracture energy as a function of AE energy from grain and corium.

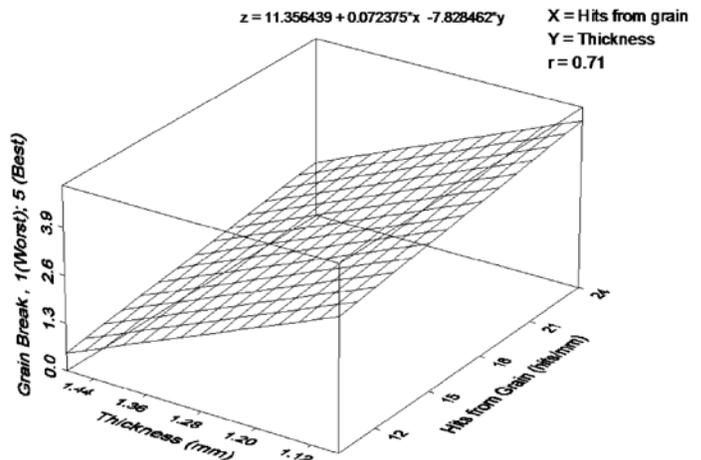


Figure 9. – Break as a function of thickness and hits from grain.

tensile deformation. It is commonly known that the higher the Young's modulus, the stiffer the leather is. Theoretically, it has been linked to the fine structure of leather such as the degree of fiber orientation^{14, 15} and fiber adhesion.¹⁶ In practical terms, this physical quantity has been associated with leather softness, temper, and handle.^{17, 18} In fact, its reciprocal has been named compliance in the literature.¹⁹ It has been known to be extremely sensitive not only to changes of composition, moisture, and fatliquor concentration, but also to various leathermaking processes such as drying and staking.

Figure 7 is a regression plot of Young's modulus as a function of AE hits from the grain and corium. It shows the Young's modulus has a positive correlation with AE hits from the corium, whereas with the grain the Young's modulus has a negative correlation. The reason for this difference is again probably due to the structural difference between the two layers. The greater Young's modulus for the corium indicates a more elastic fibrous structure, which results in more AE hits. However, for the grain, the higher Young's modulus indicates a stiffer grain, in which the sheet-like fibrous structure is not easy to deform by the sensors and therefore produces less hits.

Figure 8 is a regression plot of fracture energy as a function of AE energy from the grain and corium. Fracture energy was determined by measuring the energy required to fracture the leather sample, which is the area under the stress-strain curve.¹⁴ Figure 8 shows the fracture energy has a positive correlation with AE energy from the corium, whereas with the grain the fracture energy has a negative correlation. The reason for this difference is again ascribed to their structural difference. The higher AE energy value reflects a denser fibrous structure, which results in a greater fracture energy for the corium layer. However, this principle doesn't apply to the grain, because the denser grain structure is comprised of a rigid sheet-like fibrous structure, thereby exhibiting a negative effect on the fracture energy. Importantly, the results imply that the fracture energy of leather can be predicted as demonstrated in Figure 8.

AE Evaluation of Break

The key quality associated with the aesthetic value of leather is grain "break," also named "break of leather." It is generally characterized by the wrinkles formed on the surface of the leather when it is bent or flexed inward.²⁰ Typically, there are two types of grain break: a fine break and a coarse break. "Fine break" has many fine wrinkles per square inch, which in fact is more desirable for today's customers. Most tanners want to produce the finest break possible in their product. A "coarse break" on the other hand will have fewer wrinkles and be more pronounced, giving the grain surface a loose appearance. Poor tannery processing in the beaming, tanning and retanning or the use of stale hides, can often cause coarse

break.²⁰ Moreover, break can vary naturally across the hide, with the butt having a finer break than the shoulder and belly. Tannery processes that prevent the fibrils from sticking together tightly on the grain surface are the key factors producing fine break. We manually assessed the break of the samples using an arbitrary scale having five levels of break ranging from 5, fine (the best), to 1, coarse (the worst). Figure 9 shows that the grain break becomes finer as the grain AE hits increases. The higher AE hits is an indication of softer leather as mentioned previously; therefore, the results revealed that softer leather is prone to better grain break. Figure 9 also demonstrates that thickness has significant effects on the grain break; the thicker samples tend to have poorer grain break.

CONCLUSIONS

We have been working on developing a nondestructive AE testing method for leather. Previous reports mainly described nondestructive AE evaluation based on AE signals from either the corium or grain layers. This is the first report revealing the method using AE signals captured from both the grain and corium sides simultaneously. The developed nondestructive method is based on measuring the AE signals induced by small (recoverable) deformations of leather by pressing AE sensors against the grain and corium side of the leather. Observations showed that the tensile strength, stiffness, fracture energy, and grain break of leather could be measured nondestructively by using the AE methods described in this report. The significance of this finding is profound, especially as a quality control/quality assurance method for manufacturing and the potential for being a nondestructive testing method. The grain break of leather, which is very time-consuming to determine and often subjective between different operators, has been shown to be predictable using this nondestructive method, potentially making it more of a quantitative measurement instead of a subjective measurement between operators. Lastly the tensile strength, which could only be determined using a materials testing machine to destructively test leather, could be predicted using this nondestructive method. In conclusion, this research provides the industry with a nondestructive way in which to evaluate the quality of product without damaging the leather.

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