CHARACTERIZATION OF MECHANICAL PROPERTIES OF LEATHER WITH AIRBORNE ULTRASONICS

by

CHENG-KUNG LIU,* NICHOLAS P. LATONA, MARYANN TAYLOR, CHRISTOPHER EBLE AND MILA L. ALDEMA-RAMOS United States Department of Agriculture,** Agricultural Research Service EASTERN REGIONAL RESEARCH CENTER, 600 EAST MERMAID LANE, WYNDMOOR, PA 19038

Abstract

A nondestructive method to accurately evaluate the quality of hides and leather is urgently needed by leather and hide industries. Effects have been made to develop airborne ultrasonic (AU) testing method using non-contact transducers to evaluate the quality of hides and leather. We previously reported the research results demonstrating the AU technology for revealing defects in hides and leather that were difficult to be found during visual inspection. Recently new research was carried out to develop AU methods to nondestructively characterize the mechanical properties of leather. Observations showed a strong correlation between the mechanical properties of leather and the corresponding AU parameters based on the distribution of the transmission time (time of flight) through leather. We also used this nondestructive method to characterize the grain break of leather. Results showed the difference in grain break could be determined from the AU parameters collected from moving the AU sensors over a leather sample. Observations showed the poorer the grain break, the higher the time of flight distribution. In short, this study demonstrated that the tensile strength, stiffness, toughness and grain break could be nondestructively determined by AU.

INTRODUCTION

Airborne Ultrasonic (AU) methods have been used extensively in the inspection of lumber and composites.¹⁵ We were the first to develop AU methods to detect leather defects and characterize leather quality. In the earlier reports, we demonstrated that AU testing without direct contact with samples offers a great potential method for the nondestructive evaluation of the quality of leather.⁶⁻⁷ As this is a non-contact technique, it is an ideal characterization method for large leather or hides. The AU sensor is designed for dynamic measurements and offers several key advantages in automated and moving process applications. AU testing involves pulsing ultrasonic signals at the material and measuring the reflected amplitude of those signals emanating from the material.⁷ The amplitude of ultrasonic signals reflected at the surface of a planar material (such as films, sheets, fabrics, and leather or hides) is a function of the material's surface morphological variations. Therefore defects, such as scars, insect bites, or knife cuts should be able to be detected because they will change the intensity of the AU signal reflecting from the surface of material. Our previous studies indicated that AU testing could reveal the presence of defects in the leather or any other physical discontinuity that could affect the leather quality.

Manuscript received September 25, 2014, accepted for publication December 9, 2014.

^{*}Corresponding author e-mail: ChengKung.Liu@ars.usda.gov; Tel (215) 836-6924.

^{**}Mention of trade names or commercial products in this article is solely for the purpose of providing specific information and does not imply recommendation or endorsement by the U.S. Department of Agriculture (USDA). USDA is an equal opportunity provider and employer.

Our recent AU testing involves pulsing ultrasonic waves and measuring the amplitude of those waves transmitted through the material.⁸⁻¹⁰ We believe by using the through transmission mode, more useful information can be extracted from the AU scan than the reflective mode, particularly for hides, which are covered by hair.

When performing AU testing, AU waves must travel from air, which is a medium with low acoustic impedance, to a medium such as hides and leather with considerably higher acoustic impedance. Therefore, selection of the proper AU transducers and frequency are critical to achieve enough penetration of ultrasonic waves in order to extract important information related to the structure and properties of leather, such as the amount of defects, morphology, strength and softness. When an ultrasonic wave is passed from one medium to another (for example, from air to leather), a large portion of that energy is reflected and the remaining energy is transmitted. The physical quantity that governs the reflected and transmitted relationship is referred to as acoustic impedance (Z), which is governed by its density and elasticity.¹¹ When the acoustic impedances of the materials on both sides of the boundary are known, the fraction of the incident wave intensity that is reflected can be calculated as the acoustic reflection coefficient (R%) as described in our earlier report.¹⁰ The higher the R%, the greater the percentage of energy will be reflected at the interface or boundary between one medium and another. Therefore, it is predictable that the mismatch of acoustic impedance, as indicated by greater R%, weakens the sound wave transmission. Defects such as voids, insect damage, and brands will change R% and consequently affect the amplitude of the wave transmitted through the material, which will show up in AU images such as C-scans.

Our previous studies indicated that the key for success in AU testing is to use AU transducers with low frequencies, which leads an effective transmission of ultrasonic waves through hides or leather.⁸⁻¹⁰ The variations in the AU quantities, such as amplitude (AMP) or time of flight (TOF) were colored coded into C-scan images to reveal the location and shape of the defects or some other physical discontinuity that existed in hides. To perfect AU methods for hides and leather inspections, research was recently carried out to study the effects of transducer frequency, thickness of leather, and AU gain on the resultant AU amplitude received, which was color coded into a C-scan image.¹⁰ The research results showed the 100 KHz transducer works well for crust leather. This study also showed the AMP and TOF are strongly affected by the sample thickness and instrument gain applied to the AU tests. The results will be instrumental in finalizing the development of AU technology for the characterization of quality of leather and hides.

In this new study, our objective is to establish the correlation and model the relationship between AU quantities and leather mechanical properties. We used software to translate the C-scan of leather into numeric values that was correlated with the mechanical properties of leather. The derived regression equations could be used for the estimation of leather mechanical properties based on the AU testing, which would be useful in a more objective grading system.

EXPERIMENTAL

The samples used to study the correlation of crust leather mechanical properties to the AU properties were upholstery weight crust from shaved, 1.0-1.2 mm, wet white. The wet white was retanned, colored, and fatliquored according to the procedure previously published.¹²⁻¹³

The AU test system consisted of two ultrasonic transducers approximately 3 cm apart, a transmitter (model: NCG100-D38, the Ultran Group, State College, PA) with a diameter of 38 mm pulsed with a tone burst through a power amplifier, and a receiver (model: NCG100-D25) with a diameter of 25 mm connected to a preamplifier were mounted on a computercontrolled X-Y scanner using the software UTWIN version E1.81 (NDT Automation, Princeton Jct., NJ) that allowed the transducer/receiver array to be moved over the entire surface of the crust. The samples were clamped taut across a frame with two parallel bars in order to minimize any slack in the sample. For one AU scanning result, there are various AU quantities that can be displayed as a function of time or sample position. The velocity, amplitude, and duration of ultrasonic waves measured by the receiver changed with the material properties of test samples. The C-scan is very commonly used in AU testing, in which the transmitted AU pulses were captured and the amplitudes of the transmitted pulses were mapped using pseudo color from the maximum amplitude in gate 1 or gate 2 set on the A-scan.⁹ The A-scan presents the waveform of the received signal and gate 1 is set to the first wave and gate 2 is set to the second wave. Converting C-scan images into numeric data is the key step to enable one to quantitatively determine the correlation with other materials properties. The resolution of the scan or data collection sensitivity was set to 0.5 mm and the index or advancement of the sensor was set to 0.5 mm. Time of Flight (TOF) values were recorded in the UTWIN software, where the TOF values represent the time it took to reach the maximum peak in the defined gate or period of time from 150 to 250 µs. A distribution of the TOF values from 150 to 250 µs (denoted as $TOF_{150-250}$) was recorded in increments of 10 µs, a distribution of TOF values from 150 to 200 μ s (denoted as TOF₁₅₀₋₂₀₀) was recorded in increments of 20 µs, a TOF value from 200 to 250 µs (denoted as $\mathrm{TOF}_{\mathrm{200-250}})$ was recorded and the average TOF value (denoted as TOF_{ave}) was recorded from 150 to 250 µs.

Subjective material properties were evaluated according to those reported in a previously published paper by Taylor et al,¹²⁻¹³ where the overall value represents one evaluator's

perception of the crust mostly based on break but also includes handle, fullness, and color. A rating value from 1 to 5 was allocated, with 1 being the worst and 5 being the best. An Insight-5 test frame and Testworks-4 data acquisition software (MTS Systems Corp., Minneapolis, MN) were used throughout this work. Mechanical properties including tensile strength, elongation, Young's modulus, and fracture energy (toughness) were tested as described in a previous paper.¹⁴

RESULTS AND DISCUSSION

Figure 1 shows a 3-D regression plot of tensile strength as a function of TOF_{avg} and $\text{TOF}_{200-250}$. It shows the tensile strength has a positive correlation with TOF_{avg} , whereas with the $TOF_{200,250}$ the tensile strength has a negative correlation that is defined as a relationship between two variables in which one variable increases as the other decreases, and vice versa. The reason for this difference is ascribed to leather structural difference. The greater amount of TOF_{ave} reflects a denser fibrous structure, therefore stronger leather, which results in a longer transmission time of ultrasonic waves through leather samples. However, this principle doesn't apply to the TOF₂₀₀₋₂₅₀, probably because of the overly dense structure resulting in higher rigidity, which has a negative impact on the tensile strength. Importantly, the results demonstrated in Figure 1 imply that the tensile strength of leather can be predicted by the AU nondestructive test method described here, by measuring the TOF. 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.



Figure 1. Tensile strength as a function of $\text{TOF}_{avg}(X)$ and $\text{TOF}_{200.250}(Y)$.

Figure 2 shows a 3-D regression plot of elongation as a function of thickness and $\text{TOF}_{220-230}$. Studies showed elongation could not totally correlate with AU parameters. In fact, the thickness of leather samples has to be taken into consideration. It is reasonable to expect that the thicker sample has more volume to be stretched to the breaking point. Figure 2 shows the elongation increase with thickness, whereas with the $\text{TOF}_{220-230}$ the elongation has a negative correlation. Again, the greater $\text{TOF}_{220-230}$ value reflects a denser fibrous structure, therefore leather has a decreased elongation.

Leather Stiffness

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



Figure 2. Elongation as a function of $TOF_{220-230}(X)$ and thickness (Y).



Figure 3. Young's modulus as a function of $TOF_{200-250}(X)$ and $TOF_{210-220}(Y)$.

modulus, the stiffer the leather is. In fact, its reciprocal has been named compliance in the literature.¹⁵ Theoretically, it has been linked to the fine structure of leather, such as the degree of fiber orientation¹⁶⁻¹⁷ and fiber adhesion.¹⁸ In practical terms, this physical quantity has been associated with leather softness, temper, and handle.¹⁹⁻²⁰ 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 3 is a regression plot of Young's modulus as a function of $\text{TOF}_{200-250}$ and $\text{TOF}_{210-220}$. It shows the Young's modulus has a positive correlation with both $\text{TOF}_{200-250}$ and $\text{TOF}_{210-220}$. As mentioned before, the greater TOF value indicates a longer transmission time of ultrasonic waves through the leather samples. This reflects a denser fibrous structure, and therefore stiffer leather, which results in a higher Young's modulus.

Leather Toughness

Toughness has been described in a previous report as a physical quantity associated with the energy required to fracture leather, in material science, it is named "fracture energy." Fracture energy was determined by measuring the energy required to fracture the leather sample, which is the area under the stress-strain curve.¹⁴ Previously we have reported that contrary to tensile strength, the sampling angle shows little effect on the toughness. Good toughness reflects a superior balance of strength and flexibility as shown in a regression plot of fracture energy as a function of tensile strength and elongation.

Figure 5 shows a regression plot of fracture energy as a function of TOF_{avg} and $\text{TOF}_{200-250}$. It shows the fracture energy has a positive correlation with TOF_{avg} , whereas with the $\text{TOF}_{200-250}$ the fracture energy has a negative correlation. The reason for this difference is ascribed to their structural difference. The greater amount of TOF_{avg} reflects a denser fibrous structure, therefore tougher leather, which results in a longer transmission time of ultrasonic waves through leather samples. However, this principle doesn't apply to the $\text{TOF}_{200-250}$, probably because of overly dense structure resulting in higher rigidity, which has a negative impact on the fracture energy.

AU Evaluation of Break

Grain "break," also named "break of leather." is the key quality associated with the aesthetic value of leather. It is generally characterized by the wrinkles formed on the surface of the leather when it is bent or flexed inward.²¹ There are two types of grain break , typically: 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,

z = -3.591444 + 0.211309*x + 0.050540*y



Figure 4. Fracture energy as a function of tensile strength (X) and elongation (Y).



Figure 5. Fracture energy as a function of $TOF_{ave}(X)$ and $TOF_{200.250}(Y)$.



Figure 6. Break as a function of $\text{TOF}_{210\text{-}220}(X)$ and $\text{TOF}_{200\text{-}250}\left(Y\right)$.

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 6 shows a regression plot of break as a function of $TOF_{200-250}$ and $TOF_{210-220}$ It shows the break has a negative correlation with both $TOF_{200-250}$ and $TOF_{210-220}$. Interestingly to note that this is exactly opposite to the behavior showing for Young's modulus, in which Young's modulus has a positive correlation with both $\mathrm{TOF}_{200-250}$ and $\mathrm{TOF}_{210-220}$. The lower TOF value indicates a shorter transmission time of ultrasonic waves through the leather samples. This reflects a less dense and flexible fibrous structure, therefore the softer leather, which results in better break evaluation.

CONCLUSIONS

We have been working on developing a nondestructive AU testing method for leather. Previous reports mainly described nondestructive AU evaluation on detecting leather defects. This is a report revealing the method using AU to characterize mechanical properties. The developed nondestructive method is based on measuring the AU waves transmitted through the leather sample. Previously we used AU parameters derived from the amplitude distribution of the C-scan. However, in this investigation, we found that the distribution of Time of Flight (TOF) is a better AU parameter to correlate the mechanical properties of leather. Observations showed that the tensile strength, stiffness, fracture energy, and grain break of leather could be measured nondestructively by using the AU 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. In conclusion, this research provides the industry with a nondestructive way in which to evaluate the quality of product without damaging the leather. A knowledge gap, however, still exists for the effects of change in humidity, temperature, and scanning speed on the AU transmission through the samples. These factors will be examined in the new project to develop a commercially viable AU method to characterize the leather and hides quality.

Acknowledgement

The authors thank Dr. Seung-Chul Yoon of Russell Research Center for his invaluable suggestions. Thanks are also extended to Joe Lee for preparing leather and grading the break. Especial thanks are also extended to Lorelie Bumanlag for retanning, coloring, and fatliquoring all of the samples.

REFERENCES

- Sanabria, S. J., Mueller, C., Neuenschwander, J., Niemz, P., and Sennhauser, U.; Air-Coupled ultrasound as an accurate method for bonding assessment of glued timber. *Wood Sci Technol*, 1-15, 2010.
- Rojek, M., Stabik, J., Sokół, S.; Fatigue and ultrasonic testing of epoxy-glass composites. *Journal of Achievements in Materials and Manufacturing Engineering* 20, 183-186, 2007.
- Liu, C.-K., Latona, N. P. and Liu, L.S.; Characterizations of biobased materials using acoustic emission methods, pp.138-161. In Kubica, S., Zaikov, G. E. and Liu, L.S.(eds) Biochemical Physics and Biodeterioration New Horizons, Institute for Engineering of Polymer Materials and Dyes, Torun, Poland. 292 pp, 2012.
- Li, Weili, Coffin, D. R., Jin, T. Z., Latona, N. P., Liu, C.-K., Liu, Bo, Zhang, J., and Liu, L.S.; Biodegradable composites from polyester and sugar beet pulp with antimicrobial coating for food packaging. *J. Applied Polymer Sci.* 126, E361-E372, 2012.
- 5. Hosten, B., Castaings M., Tretout H. and Voilluame H.; Identification of composite materials elastic moduli from Lamb wave velocities measured with single sided, contactless ultrasonic method. *Rev Prog Quant Nondestruct Eval* **20**, 1023-1030, 2001.
- Liu, C.-K, Godinez-Azcuaga, V. F., Latona, N. P., Hanson, M. and Finlayson, R. D.; New acoustic methods for nondestructive evaluation of leather quality. *JALCA*. 103(3), 89-127, 2008
- Liu, C.-K, Godinez-Azcuaga, V. F., Latona, N. P., Hanson, M. and Ozevin, D.; Airborne ultrasonics for nondestructive evaluation of leather quality. *JALCA* 103(7), 209-214, 2008.
- Liu, C.-K. and Latona, N. P.; Airborne ultrasonic inspection of hides and leather. *JALCA* 106(11), 326-331, 2011.
- Liu, C.-K. Latona, N. P., and Yoon, S.-C.; Evaluation of hides, wet blue and leather using airborne ultrasonics. *JALCA* 108(4), 128-138, 2013.
- Liu, C.-K. and Latona, N. P.; Effects of thickness and gain on the amplitude of airborne ultrasonics. *JALCA* 109(3), 70-75, 2014.

- 11. Ensminger, D.; Ultrasonic, the low- and high-intensity applications. Marcel Dekker, New York, 1973. p 23.
- Taylor, M.M., Marmer, W.N., and Brown, E.M.; Effect of fillers from enzymatically modified proteins on mechanical properties of leather. *JALCA* 103(4), 128-137, 2008.
- Taylor, M.M., Lee, J., Bumanlag, L.P., Hernàndez-Balada, E., and Brown, E.M.; Treatments to enhance properties of chrome-free (wet white) leather. *JALCA* 106(2), 35-41, 2011.
- Liu, C.-K. and McClintick, M. D.; An energy approach to the characterization of the fracture resistance of leather. *JALCA* 92(5), 103-118, 1997.
- 15. Morton, W.E., and Hearle, J.W.S.; Physical Properties of Textile Fibers. The Textile Institute, Manchester and London, pp. 272-273, 1978.
- Kronick, P. L., and Buechler, P. R.; Fiber orientation in calfskin by laser light scattering or X ray diffraction and quantitative relation to mechanical Properties. *JALCA* 81 (7), 221-230, 1986.

- 17. Kronick, P. L., and Buechler, P. R.; Fibre orientation and small deformation modulus of stretched, partially dried hide. *JALCA* **83**(4), 115-124, 1988.
- Kronick P L., Page, A., and Komanowsky, M; An acoustic emission study of staking and fatliquor. *JALCA* 88(5), 178 186, 1993.
- Guy, R.; A comparison of some foot comfort properties of a full chrome side leather and 'porvair'. *BLMRA J.* 15 (3), 65 68, 1972.
- 20. Diebschlag, W.; Measurements of the elasticity of different shoe upper materials as well as their maximum pressure on the foot during walking. *Leder* **26** (1), 7 18, 1975.
- 21. Tancous, J. J.; Skin, Hide and Leather Defects. Lee Corporation, Cincinnati, pp. 13-14, 1986.