# **Differences in strength between the grain and corium layers of leather**

# D.N. O'LEARY, G.E. ATTENBURROW

*British School of Leather Technology, Nene College, Moulton Park, Northampton, NN2 7AL, UK* 

The tearing resistance of the two principal strata of leather (the grain and the corium layers) has been assessed via the measurement of tearing energy and notch sensitivity. Observations of the distribution of strain around notches and the form of the tear tips are also reported. It was found that the grain layer had only 20% of the tearing energy possessed by the corium. In addition, the strength of the grain was considerably more sensitive to the presence of sharp notches. It is suggested that these differences in strength between the two strata of leather are associated with the greater ability of the corium layer's fibre structure to impede propagating tears by means of tear tip blunting and fibre pull out.

# **1. Introduction**

The leather making process involves the chemical and mechanical manipulation of animal hides and skins to remove unwanted materials such as hair, epidermis and ground substance while leaving intact the feltwork of collagen fibres that conferred structural integrity to the skin. During leather production, the collagen macromolecules are subjected to a process of chemical crosslinking (tanning) that provides increased thermal stability and resistance to microbial attack [1].

The microscopic examination of a cross-section of bovine leather, even at low magnification, reveals two structurally distinct layers (Fig. 1). The upper layer, originally closest to the surface of the animal, is termed the grain layer and is composed of interwoven collagen fibres that are of diameter  $\lesssim 5 \mu m$  near the grain surface  $\lceil 2 \rceil$ . It is the grain layer that contains the hair follicles. The thicker corium layer, immediately beneath the grain layer, is composed of a feltwork of thicker bundles of fibres (typically of diameter  $\approx$  100 µm). The boundary between these two layers is not precise and there is a zone in which the collagen fibres progressively reduce in thickness from  $\sim$  100 µm to  $\leq$  5 µm. Leather finds wide application as a quality material for shoes, clothing, upholstery, luggage, bags, etc. It is generally accepted that the tearing resistance of leather is good; however, there are circumstances when susceptibility to tearing is a problem. For example, during the lasting (stretch forming) operation of shoe making, small cracks can occur in the grain layer  $\lceil 3 \rceil$  that are detrimental to the appearance, wear and life of the shoe. In addition, when leather is split into thin grain layers ( $\leq 1$  mm) it suffers a significant loss of tensile strength compared with full thickness leather [4]. This phenomenon has limited the application of thin leathers.

The observations noted above suggest that the grain layer is considerably weaker than the corium. However, these differences have yet to be fully characterized and specifically, no measurements of tearing energy have been reported. It is also apparent that understanding of the reasons for these differences in strength is incomplete. This present study is therefore concerned with (a) establishing the magnitude of the tearing energy of the grain and corium layers of bovine leather and (b) measuring and seeking to understand differences in notch sensitivity and tear propagation between these two strata of leather.

# **2. Experimental details**

Partially processed leather (termed wet blue) was obtained from a UK tannery. This leather was produced from cattle hides and had not been subjected to the normal dyeing and oil application (fatliquoring) processes. In this work, it was desirable to avoid the fatliquoring stage because the deposition of oils within the fibrous structure of leather is known to influence its strength [5]. During fatliquoring, uneven deposition of oil through the leather thickness is common. Therefore, potential uncertainty arises in assigning the cause of strength differences between the discrete leather strata.

Simple air drying of unfatliquored leather leads to an undesirable degree of interfibre adhesion and produces a material similar in stiffness to plywood. To avoid this situation, the leather was dried by exchanging water with acetone followed by evaporation of the acetone.

The dried leather was split through the grain-corium boundary using a band knife splitting machine. Trouser tear samples (total size 50 mm  $\times$  25 mm, leg dimensions 25 mm  $\times$  12.5 mm) were cut and tested at



*Figure l* Low magnification optical micrograph showing the two principal strata of bovine leather, the grain layer (above the dashed line) and the corium layer (below the dashed line). Length of the bar  $is 1 mm$ .

a speed of  $100 \text{ mm min}^{-1}$ . The tearing energy was calculated as follows [6]

$$
T = \frac{2F}{t} \tag{1}
$$

where  $F$  is the mean tearing force and  $t$  is the sample thickness. Four nominally identical samples were tested and a mean and standard deviation calculated.

Notch sensitivity [7] testing employed leather strips of 120 mm  $\times$  25 mm in size that contained a sharp cut in the centre of and perpendicular to one long edge (Fig. 2). The length (a) of these cuts varied from 0.5mm to 12.5mm. These notched strips were strained at a speed of  $100 \text{ mm min}^{-1}$  until failure occurred. Tear testing and notch sensitivity testing was carried out on an Instron 1122 testing-machine housed in a room at  $20^{\circ}$ C and 65% relative humidity (in which samples had been conditioned for 48 h before testing.)

To facilitate the measurement of strain distributions, an array of dots was printed onto a single edge notch specimen ( $a = 10$  mm). The array consisted of four columns of 13 dots each separated from the other by 6.5 mm (Fig. 2). Straining of these samples was visually recorded using an SVHS camera. After straining, a still image (640 by 480 pixel, 256 grey shade) was obtained at intervals of one second for each test using a PC with a video capture card. Software developed by the authors enabled calculation of the strain distributions in the leather via pixel movement of dot centres. In a subsequent experiment, closer examination of the strain distribution in the region close to the propagating tear took place. Here, a closer spaced (2 mm separation) pattern of dots was printed on the leather. The video recordings from these latter experiments also enabled a detailed examination of deformation and tearing in the region around the tear tip.

Scanning electron microscopy (SEM) of tear surfaces from edge notch samples was conducted using a Hitachi \$2500 machine operated at 10 kV.



*Figure 2* Geometry of notch sensitivity specimens. The crack length  $(a)$  varied from 0.5 mm to 12.5 mm. Also shown is the larger scale grid pattern of four columns by 13 rows of dots. This enables 12 local strain calculations in each column.

# **3. Results**

## 3.1. Tearing energy

Trouser tear testing showed that the grain layer had a tearing energy of  $13.3 \pm 1.6 \text{ kJ m}^{-2}$  whereas the corium layer had a tearing energy of  $63.8 \pm$  $4.5 \mathrm{kJ\,m}^{-2}$ .

## **3.2. Notch** sensitivity testing

Figs 3 and 4 show the dependence of the nominal breaking stress on the length of the cut (a) for the corium and grain layers, respectively. The straight lines drawn on these figures represent the notch-insensitive case in which the only effect of the cut is to reduce the nominal breaking stress proportionately to the increase  $a/W$  (where W is the width of the sample). Fig. 3 shows that, for the corium, the breaking stresses do fall slightly below the straight line implying a small degree of notch sensitivity. However, the results for the grain (Fig. 4) show that this layer is considerably more sensitive to the presence of notches (i.e. the nominal breaking stress falls more rapidly as the length of the cut increases).

## 3.3. Strain distributions around a notch

Figs 5 and 6 show the strain distributions in the region around a 10 mm notch for the corium and grain,





*Figure 3* The dependence of nominal breaking stress on notch length for the corium layer of leather. The straight line indicates the expected relationship for a completely notch insensitive material.

*Figure 4* The dependence of nominal breaking stress on notch length for the grain layer of leather. The straight line indicates the expected relationship for a completely notch insensitive material. The curved line through the data conforms to Equation 2.



*Figure 5* Strain distribution in a notched sample of the corium layer, just prior to tear propagation. The local strains are mapped onto the original grid (cf. Fig. 2) and were measured between each pair of dots along the principal axis of strain.



*Figure 6* Strain distribution in a notched sample of the grain layer, just prior to tear propagation. The local strains are mapped onto the original grid (cf. Fig. 2) and were measured between each pair of dots along the principal axis of strain.

respectively. These results were obtained with the 6.5 mm spaced dot pattern shown in Fig. 2. In both cases, the specimens were at a strain where tear propagation was imminent (at which point the nominal strain was 19% in the grain and 29% in the corium.) The figures establish that the local strain is low in regions close to the edges of the notch, However, moving away from the notch edges along columns 1 and 2, the strains increase markedly climbing to a maximum of 20% for the grain and 28% for the corium. In the region ahead of the notch, the corium shows considerably higher strains (26-28%) compared with the grain  $(15-20\%)$ . The measurement of strains from the closely spaced  $(2 \text{ mm})$  dot pattern gave results that were consistent with the 6.5 mm spaced array. However, this smaller spaced pattern allowed local strain measurements immediately ahead

of the tear tip and indicated strains of 150% in the corium and 80% in the grain.

## **3.4. Tear propagation**

Figs7 and 8 each comprise a sequence of four video frames that show the propagation of a tear from an edge notched  $(a = 10 \text{ mm})$  sample of corium and grain, respectively. The samples in these figures possessed the 2mm spaced dot pattern. Fig. 7a depicts considerable blunting of the tear tip prior to tear propagation in the corium. During tear propagation (Fig. 7b-d) a considerable degree of fibre pull out can also be seen. With the grain, the propagating tear is sharper (particularly evident in Fig. 8d) and there also appears to be less fibre pull out.



*Figure 7* Video sequence showing tear propagation from an edge notched corium sample. The nominal strains were (a) 31.2% (b) 32.0% (c) 32.8% (d) 33.0%. Images (a) and (b) show the degree of tear tip blunting. Images (c) and (d) show a high degree of fibre pull out.



*Figure 8* Video sequence showing tear propagation from an edge notched grain sample. The nominal strains were (a) 27.3% (b) 29.3% (c) 31.2% (d) 33.2%. In contrast to the results for the corium layer, the tear tip is sharper (especially noticeable in (d)).



*Figure* 9 A scanning electron micrograph of the surface of a tear propagated from an edge notch in the grain layer. Note the relative absence of fibre pull out in the upper region closest to the surface. Length of bar is  $250 \,\mu m$ .

Fig. 9 shows an SEM micrograph of the tear surface after an edge notch has propagated through the grain layer. It is apparent that the fibre pull out is not uniform throughout the thickness and that there is a layer some  $250 \mu m$  thick where the fibre pull out is minimal. This layer is adjacent to the outer surface of the grain (i.e. opposite the surface that was originally in contact with the corium layer.)

#### **4. Discussion**

#### 4.1. Notch **sensitivity**

It is seen that the nominal breaking stress of the grain layer of leather displays a considerable degree of notch sensitivity. Indeed, the data accords with a relationship of the form:

$$
\sigma_{\rm f} = K a^{-1/2} \tag{2}
$$

where  $\sigma_f$  is the nominal fracture stress, K is a constant and a is the notch length. This relationship is indicated by the curve drawn through the points in Fig. 4. Equation 2 is of course the relationship between breaking stress and crack length predicted by Griffith's energy balance approach to fracture in linearly elastic materials [8]. Measurements of the tensile properties of the grain layer show it is neither linear nor perfectly elastic and so conformity of the data to Equation 2 is surprising.

In contrast to the grain layer, the nominal breaking stress of the corium is much less sensitive to the presence of notches and it is interesting to consider how this phenomenon may be explained. The notch sensitivity of non-linear materials has been considered by Purslow [7]. His discussion implies that notch insensitive materials have an area to the left of a vertical line through the tip of the notch, which is free of strain energy (Fig. 2b [7]). In contrast a notch sensitive material has a strain-energy-free zone confined to a semicircular region around the notch (Fig. 2a [7]).

However, despite marked differences in notch sensitivity, it is apparent that the grain and corium have similar strain distributions around an edge notch

(cf. Figs 5 and 6.) In both layers, a region effectively free from strain energy can be distinguished close to the edges of the notch. However, away from the notch, significant local strains do exist to the left of a vertical line through the notch tip. These observations imply that differences in notch sensitivity between grain and corium are not explicable through differences in strain-energy-free zones. This does not invalidate Purslow's analysis which was developed for homogeneous "elastic" materials.

A more appropriate explanation of the differences in notch sensitivity may be based on the observed differences in crack tip configuration. It is clear from Fig. 7 that the corium layer exhibits considerable rounding of the crack tip prior to fracture. Crack tip blunting mechanisms also exist in composite materials and are known to lead to notch insensitivity [9].

Our observations of crack propagation in the grain layer (Fig. 8) show that, during propagation (where the characteristic crack tip radius is independent of the initiating stress raiser [10]) the tear tip is far sharper than the corresponding situation in the corium. Although the grain layer is a non-linear viscoelastic material, it is reasonable to assume that, as with linear elastic materials, a sharp notch will produce a significant concentration of stress at its tip with a consequent reduction in the overall fracture stress.

## **4.2. Tear** testing

The tearing energy of the corium layer, at  $64 \mathrm{kJ\,m^{-2}}$ , is approaching five times that of the grain layer and may be compared with high density polyethylene  $(T = 33 \text{ kJ m}^{-2})$  and polyvinylchloride [11]  $(T =$ 45 kJ m<sup>-2</sup>), rat skin [12] ( $T_{\text{max}}$  = 30 kJ m<sup>-2</sup>) and unfilled natural rubber [13]  $(T = 37 \text{ kJ m}^{-2})$ .

Why is the grain layer intrinsically weaker than the corium? It is doubtful that this weakness can be directly attributed to the smaller sized collagen fibres present in the grain. In studying the strength of fibres teased from leather, Morgan [14J concluded that smaller diameter fibres were somewhat stronger than those of a larger diameter.

One factor that may be implicated in the lower tear strength of the grain layer is the presence of hair follicles that originally contained the hair shaft and root [3]. Analysis of optical micrographs of the leather studied has shown that the mean surface diameter of the follicles was  $0.23 \pm 0.08$  mm and that they occupied 10% of the total grain surface. Since, after leathermaking, the follicles are effectively holes puncturing the grain layer it is reasonable to assume that the grain is weaker due to their presence. However, we do not believe that the existence of the hair follicles is the sole reason for weakness in the grain layer. Indeed, our observation of tears propagating from a notched edge show that growing tears tend to avoid the follicles, possibly due to fibres being oriented around their circumference and functioning as reinforcements. It seems likely therefore that important reasons for the relative weakness of the grain layer are (a) the inability of the structure to deform in a way that blunts tears and (b) the relatively low amount of fibre

pull out that occurs during tear propagation (cf. Figs 7 and 8). It is reasonable to assume that more energy is consumed in pulling fibres, from the entangled mass of fibres, in the corium layer and that this phenomenon contributes to its greater tearing energy. This situation is analogous to synthetic fibre composites where the degree of fibre pull out from the matrix is regarded as an important factor controlling the strength of the composite [9].

In considering the observed tear propagation in the grain layer (Fig. 8), it is important to note that Fig. 9 illustrates the existence of a sub-layer in the grain, closest to the outer surface, that is effectively free from fibre pull out. In the video sequence of Fig. 8, it is the outer surface of the grain that is being viewed. It may reasonably be assumed that the sharp tear tip is formed within this surface sub-layer with the fibre pull out occurring in the under layer that was originally attached to the corium.

# **Acknowledgements**

The authors wish to thank Mr A. Landmann, Ms A. Bugby and BLC, The Leather Technology Centre for assistance with band knife splitting and Scanning Electron Microscopy.

#### **References**

- 1. E. HE[DEMANN, in "Fibrous Proteins", edited by D. A. D. Parry and L. K. Creamer (Academic Press, London, 1979) p. 231.
- 2. M. DEMSEY, *J. Amer. Leather Chem. Assoc.* 63 (1968) 666.
- 3. L.G. HOLE, B. S. TUCK-MARTIN and J. HALE, *J. Soc. Leather TechnoL Chem.* 63 (1983) 98.
- 4. M. MAESER and O. J. DION, *J. Amer. Leather Chem. Assoc.*  49 (1954) 262.
- 5. V. MATTEI and W. T. RODDY, *ibid.* 52 (1957) 110.
- 6. i. M. WARD, "Mechanical Properties of Solid Polymers" (John Wiley, Chichester, 1983) p. 449.
- 7. P. PURSLOW, *J. Mater. ScL* 26 (1991) 4468.
- 8. A.A. GRIFFITH, *Phil. Trans. R. Soc. London,* A221 (1921) 163.
- 9. A. KELLY and N. H. MACMILLAN, "Strong Solids" (Clarendon Press, Oxford, 1986) Ch. 6.
- 10. A.G. THOMAS, *J. Polym. Sci.* 31 (1958) 467.
- 11. K. KENDALL, *J. Mater. Sci.* 14 (1979) 1257.
- 12. P.P. PURSLOW, *ibid.* 18 (1983) 3591.
- 13. R.S. RIVLIN and A. G. THOMAS, *J. Polym. Sci.* 10 (1953) 291.
- 14. F.R. MORGAN, *J, Amer. Leather Chem. Assoc. 55*  (1960) 4.

*Received 25 April and accepted 19 September 1995*