



Microscopic observation of tensile testing on leather fibre bundles

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The simultaneous acquisition of data on tensile force and extension values, as well as sequences of microscopic images, enables the process of fibre extension and rupture to be optically traced and documented.

This information reveals the way that the fibres crack. For example, it can show if either a cracking or a twisting of the sub-units occurs during the breaking process. Such information can be subsequently used to gain insight into ways to possibly improve behaviour, and within material modelling.

The fibre bundles tested were extracted from different leather types (automotive and upholstery leather) out of the axillae region of the bovine hide. From this soft and flexible leather area, fibre bundle segments of up to 20 mm length could be obtained.

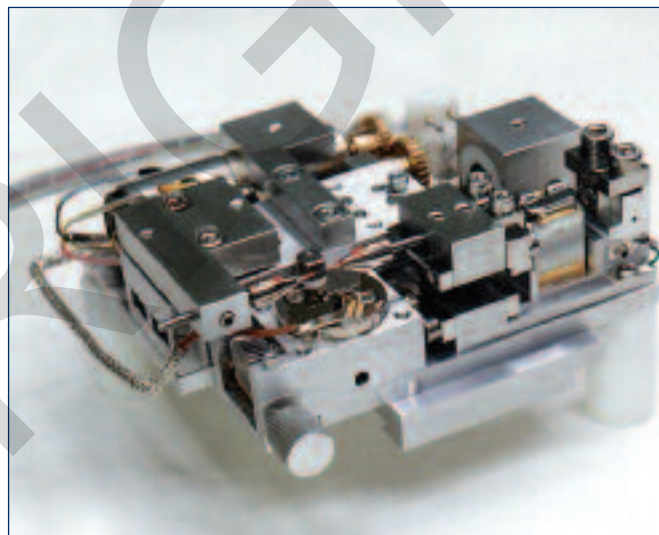
Within this research project, a microfibre tensile module manufactured by Kammrath & Weiss was used. This instrument enables the measurement of forces in the μN range. It is also vacuum-compatible, and allows both static and dynamic measurements to be made on the most sensitive test specimens. This apparatus is shown in *Panel 1*.

In conjunction with these measurements, both a scanning electron microscope (SEM) and a light microscope were used, and the following details are given:

1. Light microscopy images were obtained using a stereo microscope (GSZ 2T of Askania Mikroskop Technik Rathenow). This instrument was configured in a way to enable observations of the behaviour of the individual leather fibres under test when using the microfibre tensile module. A digital camera technique was employed, and this allowed the recording of both single images and video sequences. However, the image quality was limited by the resolution of the light microscope as this limited depth of sharpness.
2. The leather fibres as tested do not exhibit electrical conductivity, and if conventional scanning electron microscopes are used, the samples are prepared by sputtering with conductive materials.

Two FEI Electron Optics scanning electron microscopes were used. One was a model XL 30 ESEM, the second a QUANTA FEG 250. These allow testing in rarefied gases, where charging of fibres that are not electrically conductive is strongly reduced. This is particularly important because the leather fibres were not previously metallised by sputtering and were constantly uncovered during the test. In this situation the tests were performed in a water vapour atmosphere with a pressure in the range of 40 to 100 Pa.

3. The method of fixation of the fibres within the apparatus as recommended by the provider of the fibre tensile module was to use sealing wax. However, this proved to be of limited suitability as the high temperatures required in heating and melting the wax caused detrimental effects to the fibres under test.



This fixation was achieved after a preliminary fixation of the bundles to shouldered bars using instant adhesive. When performing the test using the SEM, the part of the fibre bundle between the shouldered bars was checked for being adhesive-free. This avoided any false measurements caused by an artificial stiffening of the fibres due to the adhesive.

The free length of the individual fibre bundles (fixation length) in the tensile test was approximately 4mm.

Findings from the investigation

The bonding of the fibre bundles using instant adhesive proved to be a much better technique than using sealing wax as recommended by the manufacturer of the fibre tensile module, where approximately 30% of the data gained in the test had to be rejected. During these tests many force-extension graphs showed distinct interruptions in the course. These could not be explained by processes at the level of fibres or fibrils (sliding and rupture) and had to be attributed to the bonding.

The microscopic observation of the behaviour of the leather fibres during test proved to be difficult. As the rupture position is not predictable, the whole fibre bundle segment between the fixing points had to be examined under the light microscope. For this reason only low magnification could be used.

In the first set of investigations the fibre was extracted from the axillae area of an undyed bovine leather that was metal free. The free length between the fixing points was in this case 3.94mm



and the mean fibre thickness was 175 μm ; the sequence of the tensile test under the light microscope is shown in *Panel 2*.

The images show the sequence of the behaviour of the fibres where:

Stage (a) shows the starting position

Stage (b₁) and (b₂) shows the different rupture stages

Stage (c) shows complete rupture

Stage (d) shows stage (c) with higher magnification (tension direction to the left, tension rate 10 $\mu\text{m/s}$)

Panel 2: Sequence of the video recorded during the tensile test under the light microscope

Stage (a)



Stage (b₁)



Stage (b₂)



Stage (c)



Stage (d)

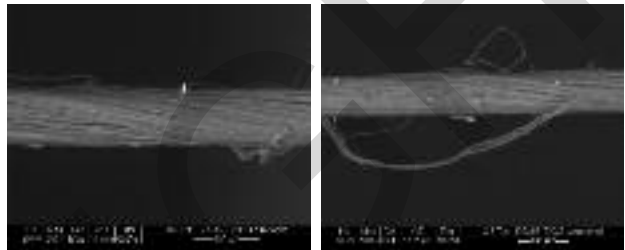


During the scanning, the electron microscope display not only obtained overview images, but also detailed images of the fibre bundles were recorded. For these more detailed images the tensile test was stopped briefly and observed to find the possible point of rupture.

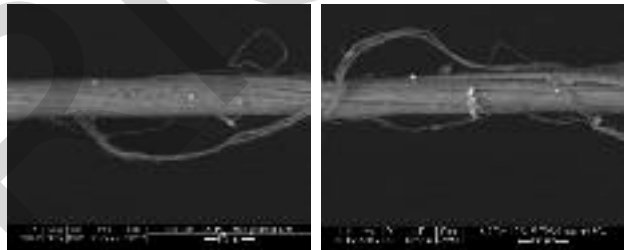
The more modern device type QUANTA FEG 250 allowed a very fine approach to the positions of interest during the test. The sequence of images showing a fibre bundle segment under increasing tensile load up to material failure is given in *Panel 3*:

Panel 3: SEM image sequences of an *in situ* tensile test

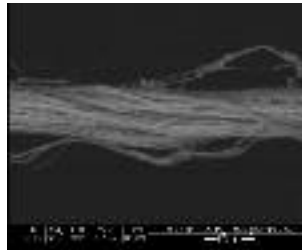
Stage (a)



Stage (b)



Stage (c)



Stage (d)



The images show the sequence of the behaviour of the fibres where:

Stage (a) shows the starting position (initial tension 70 mN), two different image details.

Stage (b) shows 400 μm and 600 μm traverse path at 10 % and 15% strain respectively.

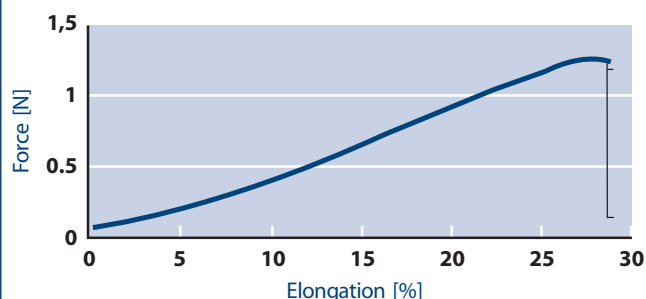
Stage (c) shows the same detail as (b) after rupture of fibre bundle occurred.

Stage (d) shows the point of rupture.



The force-elongation diagram of the tensile test documented in Panel 3 is shown in *Panel 4*:

Panel 4: Force-elongation diagram of the tensile test of a metal-free tanned leather fibre bundle.
(Tension rate 10µm/s)



Under the impact of tensile load – as shown in the first section of the diagram – the fibre crimp that was first observed disappeared. Next, the fibres increasingly oriented themselves parallel to the direction of the force applied. This region is characterised by a lower slope of force-elongation diagram.

The straightening and “bringing together” of the fibres resulted in a decrease of the bundle cross-section. As expected, fibres that were not completely fixed by the bonding technique used emerged from the bundle and thus did not align themselves.

With the further increase of the load, the fibres became more tensioned and stretched respectively until breaking of the fibre bundle. Accordingly, in the second section of the graph given in *Panel 4* shows a steeper slope.

In the final rupture phase the fibres of the bundle break individually, or several simultaneously, as soon as the load exceeds the tensile strength of the corresponding fibre. These investigations confirm the findings and observations described in specialist literature^(2, 3).

Findings from the investigation

By using a microfibre tensile module it was possible to visualise and document processes that occur at the fibre bundle level. The force-elongation diagrams of fibre bundles that were produced showed the same course and behaviour as for leather.

The orientation of the fibre structure in leather under load at fibre bundle level corresponded with a simultaneous loss of individual fibre crimping and an intensification of the arrangement of the subunits (individual fibres). Only after that stage were the fibres stressed and stretched till break.

The equipment and procedures developed in this study have provided a technique that can be used in further investigations into the modelling and rheology of leather. ☺

Acknowledgement:

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Literature:

- [1] <http://www.kammrath-weiss.com>
- [2] Martin Tuckermann, *Struktur-Eigenschafts-Beziehungen in Leder und Kollagen-Modellsystemen*. VDI Verlag GmbH, Düsseldorf, 2001
- [3] Attenburrow, G. E., *The Rheology of Leather – A Review*, *Journal of the Society of Leather Technologists and Chemists*. Vol 77, 107 – 114

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