

Corrected or non-corrected grain? FILK-QMA-1201 — a method to evaluate finished leather

Haiko Schulz, Michael Meyer

FILK – Forschungsinstitut für Leder und Kunststoffbahnen, Germany

A new method is described and illustrated, which enables differentiation between finished leathers that have been corrected or not. The method is very sensitive, and can readily detect leathers that have only been lightly corrected, even when the leathers have been slightly snuffed.

The main objective of grain correction of leathers is to remove surface defects. If the defects are slight, then buffing can be very light, so that only a small amount of the grain is removed by abrasion. The deeper the grain defects, the heavier the level of correction, and the greater the abrasion. This difference between full grain and corrected grain leathers, or the level of correction, can be seen most clearly before leather finishing.

During finishing, the highest quality full grain leathers are normally lightly finished, so that the quality of the grain is both enhanced and protected. However, heavier finishes are used to create a more uniform surface, or to attain very high physical specifications. In many instances, the finishing process also involves printing or embossing procedures that both mask surface defects and create new grain patterns.

As the level of finishing is increased, it becomes more difficult to differentiate between leathers that retain a full grain, and those that have been corrected. A finish thicker than about 10µm makes the detection of corrections impossible. And within the levels of correction, the differences between light and heavy correction—and the levels of grain damage that might lie beneath the finish—become difficult to assess.

Up to now there has been no reliable

method with which to verify this grain correction or buffing. The leather sample or finish was usually treated with different solvents, then by careful rubbing, the finish was removed from the substrate. The samples were then investigated by microscopic methods.

Because of the strong adhesion of the finish to the grain, especially of buffed leathers, it was often impossible to remove the finish without the destruction of the surface of the leather. In this event it was often impossible to differentiate whether this surface damage was a result of the method of investigation or the buffing operation.

This situation was unsatisfactory for the test laboratories, the tanneries, and their customers.

Panel I

Definitions⁽¹⁾

FULL GRAIN LEATHER

Leather bearing the original grain surface as exposed by removal of the epidermis and with none of the surface removed by buffing, snuffing or splitting.

CORRECTED GRAIN LEATHER

Leather from which the grain layer has been partially removed by buffing to a depth governed by the condition of the raw material and upon which a new surface has been built by various finishes.

The method of evaluation

An investigation was undertaken to find a method for the non-destructive removal of the finish from the grain, and to accurately assess the degree of correction of a given leather surface. To achieve these requirements, the following technique was developed.

SAMPLE PREPARATION

Sample preparation is the most important step within this type of investigation, as the finish must be removed from the surface without damaging the grain. In addition, no

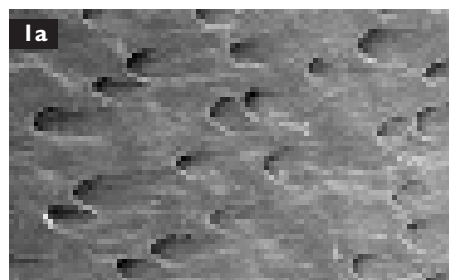
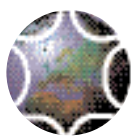


Figure 1a: Leather surface after complete removal of the finish layer. (View 150x magnification)

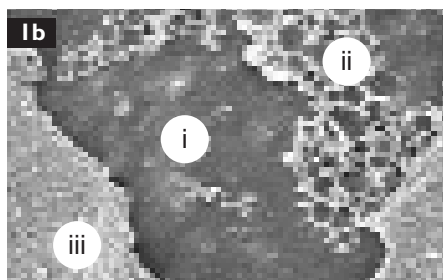


Figure 1b: Leather surface after incomplete removal of the finish layer: (i) original finish surface (ii) foam layer (iii) base coat. (View 200x magnification)

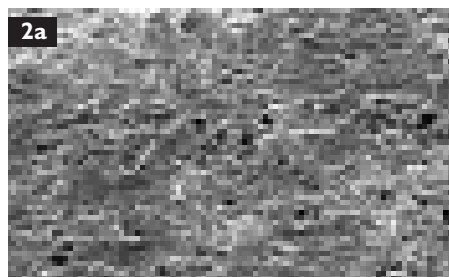


Figure 2a: Back-side of the finish layer after removal from a corrected grain leather. No fibres remaining in the finish. (View 200x magnification)

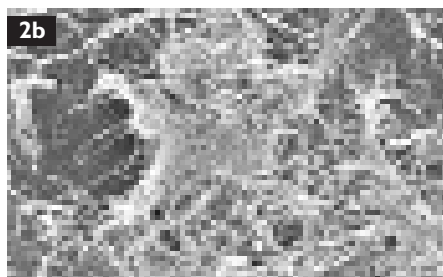


Figure 2b: Back-side of the finish layer after removal from a corrected grain leather. Fibres left sticking in the finish. (View 200x magnification)

loosely bound leather fibres should remain on the finish.

To remove the finish, the finish surface is bonded onto a carrier with a flat hard surface (such as a steel strip), which is lightly coated with cyanoacrylate adhesive according to EN ISO 11644⁽²⁾. After fixing the sample, the leather is saturated with solvent from the flesh side. Suitable solvents are mixtures of equal parts of either:

- 1 molar hydrochloric acid and methanol
- formic acid and methanol
- hydrochloric acid and acetone.

After solvent penetration, the leather is slowly peeled away from the carrier. The time of treatment, and the speed and angle of peel, are dependent on the sample (it is noted that acetone often leads to a separation of the finishing layers). This step also depends on the experience and the skill of the operator. After the removal of the leather, the samples have to be dried because fine surface structures are not visible when the samples are wet.

The base coat is often of low viscosity when applied. It therefore penetrates readily into the fibre structure of the surface, especially in areas that are damaged. In these cases the finish removal is not easy, often leading to finish-residues remaining on the grain surface, or fibres being stuck together.

To overcome this contamination, the complete sample (bonded to the carrier) is soaked in a solution of 1% Alcalase 2.4 FG (Novozyme) at 50°C for several hours. When the protein fraction has completely digested, the finish remains, showing a mirror image of the grain surface.

To evaluate the cross sections, the whole samples are cut without removal of the finish. It is important to include cross sections of the hair follicles. Three samples as a minimum are prepared from each leather to minimise the influences of localised defects or variations in hide structure.

SAMPLE EVALUATION

The evaluation comprises:

- Assessment of the leather surface after removal of the finish
- Study of the cross sections
- Observation of the back-side of the finish
- Observation of the back-side of the finish after enzymatic digestion if necessary.

For the evaluation, a light microscope with high resolution or a scanning electron microscope (SEM) is required. All images shown as figures are taken with a SEM Philips XL30 using the ESEM mode.

Before assessment, it must be ensured that the finish has been removed completely from the grain. To check this, the surface is examined by microscope to see if any finish residues remain on the leather grain (Figure 1a & 1b).

It then has to be verified that no leather fibres remain stuck to the back-side of the finish. If this is so the fibres or fibre bundles can be easily seen (Figure 2a & 2b).

In these situations, samples shown as Figures 1b and 2b must be discarded and new samples prepared.

MICROSCOPIC ANALYSIS OF THE GRAIN

Experience shows that it is not sufficient to investigate the surface of the leather, the back-side of the finish, or the cross sections alone. All the images must be studied together.

When examining the surface of the leather, both the general appearance and the status of the hair follicles are assessed. Where the leather is full grain, the openings of the hair follicles—and especially the sickle-shaped pellicles—can be clearly observed and should be without any damage (Figure 3).

Where the surface has been buffed, the structure of the grain has suffered damage, and single leather fibres and fibre bundles can be observed. The openings of the hair follicles are also damaged in varying degrees depending upon the intensity of the buffing treatment. The sickle-shaped pellicles are usually severely damaged. One can often observe the fine structure of the grain or cut/grind marks in deeper regions of the hair follicles. (Figures 4, 5 and 6).

Even when using very fine abrasion, damage can be clearly observed. This is illustrated where crust leathers were lightly abraded using fine (1200 grit) buffing paper (Figure 7).

Other marks may be found that result from other forms of

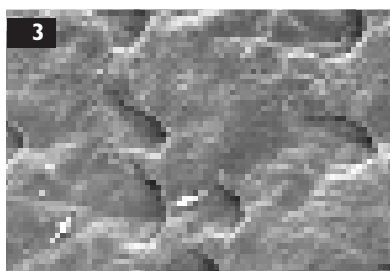


Figure 3: Leather surface of a full grain leather after successful removal of the finish. The arrows indicate the damage-free sickle-shaped pellicles and the fine structure. (View 200x magnification)

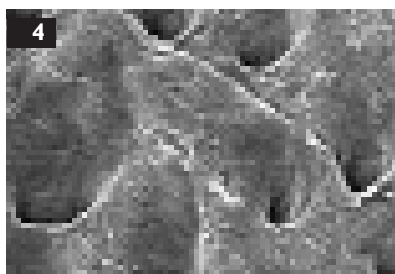
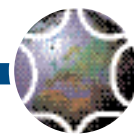


Figure 4: Surface of a lightly snuffed leather after removal of the finish. Parallel cut/grind marks are a clear characteristic of correction. (View 250x magnification)

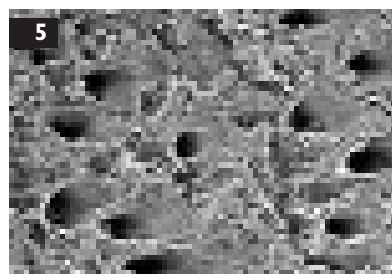


Figure 5: Surface of a buffed leather after removal of the finish. Truncated hair openings are observed as well as grind marks. (View 200x magnification)

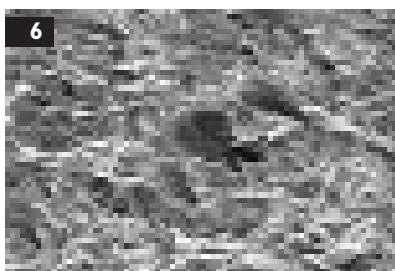


Figure 6: Surface of a heavily buffed leather after removal of the finish. Hair openings disappear in a surface with a suede-like effect. (View 200x magnification)

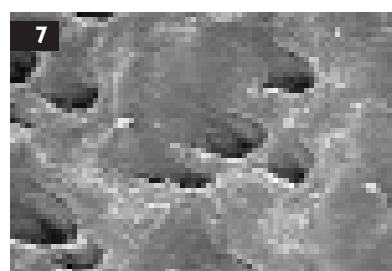


Figure 7: Surface of a crust leather after light abrasion with buffing paper (grit size 1200). Fine cut/grind marks can still be clearly observed. (View 200x magnification)

mechanical treatment. These may be similar to grind marks, but are only local and often do not show the typical parallel damage.

MICROSCOPIC ANALYSIS OF THE BACK-SIDE OF THE FINISH

The microscopic assessment of the back-side of the finish is a useful complement to a grain investigation. The fine structure of the grain often appears better on the back-side of the finish than on the grain itself. Finish that has penetrated the hair follicles is almost completely removed during sample preparation. (Figure 8).

When studying finishes obtained from corrected leathers, the hair follicles are scarcely visible. The buffing or grind marks can, however, be clearly observed (Figure 9). In addition to this, impressions of fibre bundles on the surface of the leather, agglutinated with finish can also be seen (Figure 10).

In some cases, especially when the base coat had a low viscosity, the finish cannot be completely removed by solvent as described. In this situation the leather substrate can be digested by enzymatic treatment, the back-side of the finish remaining as a mirror image of the leather surface. The leather shown as Figure 11 had not only been buffed, but also stuccoed.

Neither the finish nor the stuccoing mass is affected by enzymatic digestion.

MICROSCOPIC ANALYSIS OF THE CROSS-SECTION

The microscopic investigation of the cross section begins with the measurement of the whole thickness of the leather, the thickness of the grain, and that of the finish. A thin grain and a thick finishing layer can be the first hint pointing to a corrected surface. However, the most important criteria are the boundary layer between finish and grain, and the opening of the hair follicles.

Non-corrected leathers show a clear boundary layer. Finish and grain can clearly be distinguished and the compact layer of the grain is intact. Furthermore, the openings of the hair follicles show a complete curvature (Figure 12).

If the leather is corrected, the top of the grain is removed and there is often penetration of the finish into the grain. The openings of the hair follicles do not show any curvature, but a tapered hair channel as a result of material loss due to buffing (Figure 13).

If the grain had been damaged accidentally, such as by

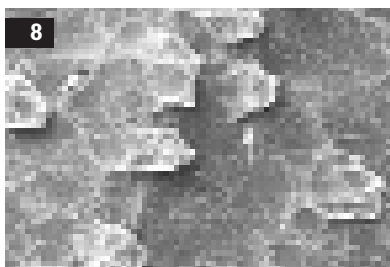


Figure 8: Back-side of a finish from a full grain leather. The mirror image of the typical grain surface is clearly observed as a result of the base coat flowing into the hair follicles. (View 200x magnification)

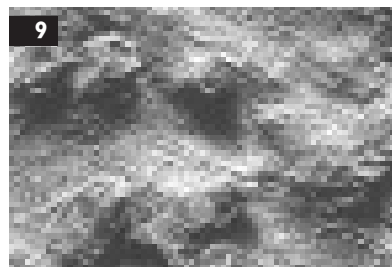


Figure 9: Back-side of a finish from a leather that was slightly corrected. The cut/grind marks are clearly visible. (View 250x magnification)

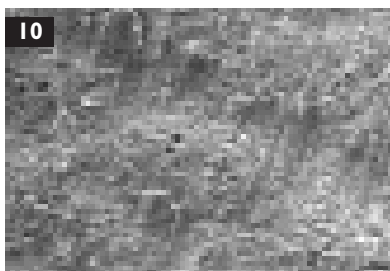


Figure 10: Back-side of a finish from a corrected grain leather. The cut/grind marks are clearly visible as well as the impression of leather fibres that remained adhering to the finish. (View 200x magnification)

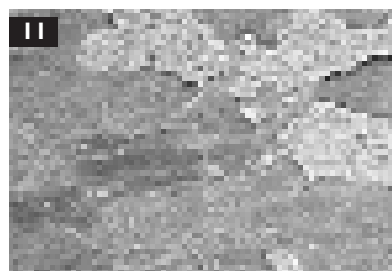


Figure 11: Back-side of a finish from a leather that was stuccoed and corrected. The leather substrate was digested by enzymatic treatment. (View 150x magnification)

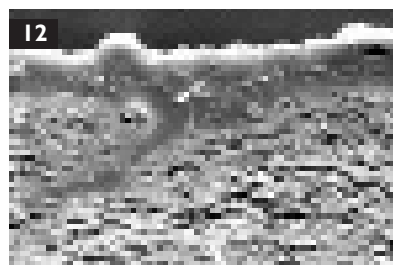


Figure 12: Cross-section of a leather that was not corrected. The curvature of the hair openings is completely preserved. (View 1000x magnification)

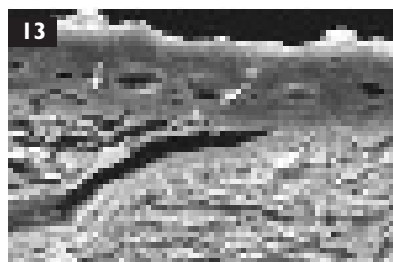


Figure 13: Cross-section of a corrected leather. The curvature of the hair openings has been lost. They appear truncated, ending at the boundary layer in an acute angle. Base coat penetration into the fibre network. (View 1000x magnification)

mechanical damage other than buffing, defects will be visible in some areas, but absent in other parts. Furthermore, the curvatures of the openings of the hair follicles can usually be found in these areas.

As a result of the complete evaluation procedure one gets the

statement “full grain” when no damages of the grain can be observed or “not full grain” when the grain is damaged. The reason for the damage can be buffing or snuffing but also mechanical damages because of bad raw materials or erroneous process control.

Conclusions

The procedure of sample preparation and microscopic investigation described has proved very sensitive in distinguishing between corrected and non-corrected leathers. An important prerequisite for an accurate assessment is the complete removal of the finish from the grain. According to the procedure FILK-QMA-1201, the combination of information from the leather grain, the back-side of the finish, and the cross-sections enable a clear differentiation between full grain leathers, and leathers that have been lightly or heavily corrected before finishing.

Acknowledgements

We thank Elisabeth Weißflog for her patience during sample preparation and Rita Mühlbach for her assistance on the enzymatic digestion of samples.

Literature

1. *International Glossary of Leather Terms*, Second edition, ICT.
2. *EN ISO 11644 “Leather—Test for adhesion of finish”*.