Characterization of Leather Structure via Metallographic Sample Preparation

by

Huayong Zhang,[1,2] Yongmei Xia,[1] Lei Shi[2] and Tianduo Li[1,2]*

[1]*School of Chemical and Material Engineering, Jiangnan University,*
Wuxi, China, 214122

[2]*Shandong Provincial Key Laboratory of Fine Chemicals,*
Qilu University of Technology, Jinan, China, 250353

Abstract

In this paper, the leather cross-sectional images based on metallographic sample preparation were introduced, including those of the vertical cross section of the grain layer and reticular layer, the fine fiber cross section and the black outlines surrounding the fiber bundles sections etc. The differences between the images of fiber bundles sections obtained by the light transmission mode and by the light reflective mode were explained. With the introduction and explain of this article, the images obtained by metallographic sample preparation can be well understood.

Introduction

The research of leather structure began in 1908. With optical microscope, the basic organization of the rawhide or leather, the woven structure of collagen fiber bundles (diameter about 60-200 um) and the loose situation of primitive fibers in the collagen fiber bundle (diameter about 5 um) were often observed.[1-5] The multiple and unique characteristics of finished leather vary considerably depending upon the leather structure. According to the observation on the finished leather structure, the finishing defects and the effects of the leather chemicals on leather can be found, so as to provide evidence for the scheme of the technological process and the selection of leather chemicals, and lay foundation for improving the quality of the finished product leather.[6] The conventional way to get the section images of leather is through observing the leather slice, which acquired by microtomy.[7-10] However, it is very difficult to obtain a complete series of section images for the reconstruction of the 3D structure of leather technically through microtomy technique. In order to solve the problems above, a new metallographic technique for revealing the true leather structures has been developed by our group[11] recently. Through this technique, a series of the optical micrographs of leather cross section were readily obtained. Since there is a lack of reports about leather section images acquired by metallographic technique, in this paper the leather structure of those section images acquired by metallographic technique was illustrated in detail. And it can pave a way and lay the foundation for further research to leather structure, for collection fiber parameters in cross section and for 3D leather network reconstruction.

*Correspondence author e-mail: litianduo@163.com
Manuscript received August 1, 2013, accepted for publication September 27, 2013.

EXPERIMENTAL

Principle

In the crossing-section, the pure resin and the fiber bundles perpendicular, inclined, and even parallel to the observational section (for simplicity, the mean of perpendicular, inclined or parallel is referred to hereafter all has the observational section as a reference) exhibit different properties such as hardness. The Vickers hardness of pure resin section is the highest, and hardness of a section of the vertical fiber bundle is higher than that of the parallel fiber bundle section (table I). So after polishing, different composition has different luminance and different light reflection effect, and has obvious interface. By a microscope with reflected light mode, a series of images with clear boundaries of the fiber bundles were obtained; the fiber bundles, which were extended to different directions, can be distinguished easily.

TABLE I
Hardness of different part of the section.

Part	Resin Section	Vertical Fiber Bundle Section	Parallel Fiber Bundle Section
HV(kg/mm^2)	36.2	11.9	6.6

Experimental Materials and Instruments

Crust leathers (dried piece of bluestock) of chrome-tanned cattle hide, obtained from Shandong Dexin Leather Company was chosen for this work (for simplicity, it is referred to hereafter as leather). The embedding medium was epoxy resin. The grade of epoxy and its hardener were E-51 and 593#, respectively, made by Jinan TianMao Resin Company, China. The mass ratio of epoxy/hardener was 4/1 for the preparation of all the samples OLYMPUS BX51 optical microscope, made in Japan, has transmitted and reflected light mode to observe samples.

Metallographic Sample Preparation Steps[11]

Mold preparation — Embedding — Mounting — Coarse Grinding — Fine Grinding — Polishing — Microscopic Examination.

RESULTS AND DISCUSSION

The images of the leather sections were recorded with the optical Microscope using light reflection model. The followings were the analysis and interpretation of the leather or fiber section images by Metallographic sample preparation.

Panorama of Leather Vertical Cross Section

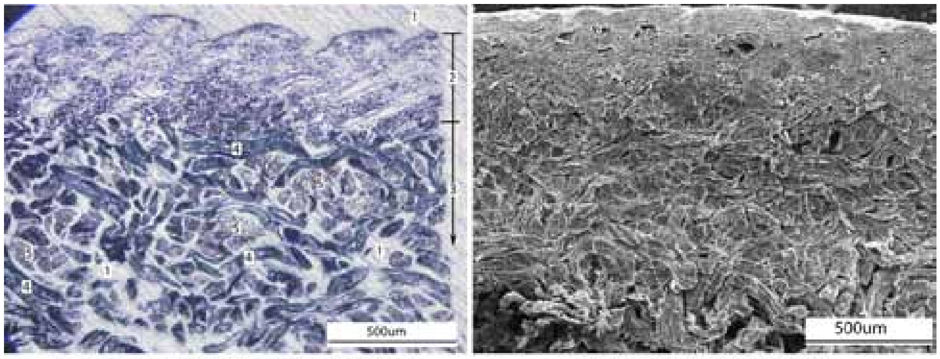
As seen in Figure 1 (a): grain layer and reticular layer can be easily identified due to the nature of their structures. The fiber bundles in the grain layer are fine and weave compactly, while the fiber bundles in the reticular layer are hair chested and weave in a crisscross fashion and forms a three-dimensional net structure. Because the fibers were pre-impregnated into the resin, fiber morphology and the spacing between fibers or fiber bundles would not change, as well as the leather nature section. Although the section of the fibers were smooth and flat via Metallographic sample preparation, clear boundaries of the fibers can be easily found and extracted accurately in the subsequent analysis due to the obvious color differences between fibers and resin under an optical microscope. The images of leather cross-section without the embedding process were taken by scanning electron microscopy (SEM) (Figure 1 (b)). In Figure 1 (b), the fiber bundles at the section appeared fluffy and the spacing between fibers was smaller than that in figure 1 (a). The deformation of the fiber bundles, especially those parallel to the cross sectional direction, can be observed, and the boundary of fiber bundles cannot be distinguished readily. The fibers in the stereoscopic SEM images display overlapping and intermingling, resulting in the complications and difficulties in extracting the boundaries of fiber bundle sections.

Presentation Mode of the Grain Layer Vertical Cross Section

Figure 2 is the cross section images of two kinds of grain layer at the different magnifications. A section running perpendicular to the fibers shows fibers to have an irregular round outline or oval face or dot, and the fibers parallel to section plane show as curved dotted lines. Some of fiber having a range of 2-6 microns in diameter, which can be regarded as a single fine fiber form, was not gathered into a bundle. The fibers wove tightly, creating a fine and compact interweaving natural cover. Figure 2 (f) showed irregular circles enclosed by a compact interweaving fine fibers layer, resulting from the traces of hair follicles and various gland in the grain layer. Sample 1 has larger spacing between the fibers and more obvious papillae than sample 2, resulting that sample1 is softer and more loosen than sample 2, while the surface of sample 2 is smoother.

Presentation Mode of the Reticular Layer Vertical Cross Section

Figure 3 is the vertical cross section of reticular layer. The fibers in the reticular layer are gathered into bundle whose diameter is from 20-160um. The bundles all have distinct outline, no standard shape, and have certain gaps between them. It is very easy to distinguish the fiber bundle, which is perpendicular to the section from which is parallel to the section by shape and length.



(a) By Metallographic sample preparation (b) Cut directly and by SEM

Figure 1. The microscopic images of vertical cross-section of leather.
(1 — epoxy resin, 2 — grain layer (papillary dermis), 3 — reticular layer,
4 — parallel fiber bundles, 5 — perpendicular fiber bundles.)

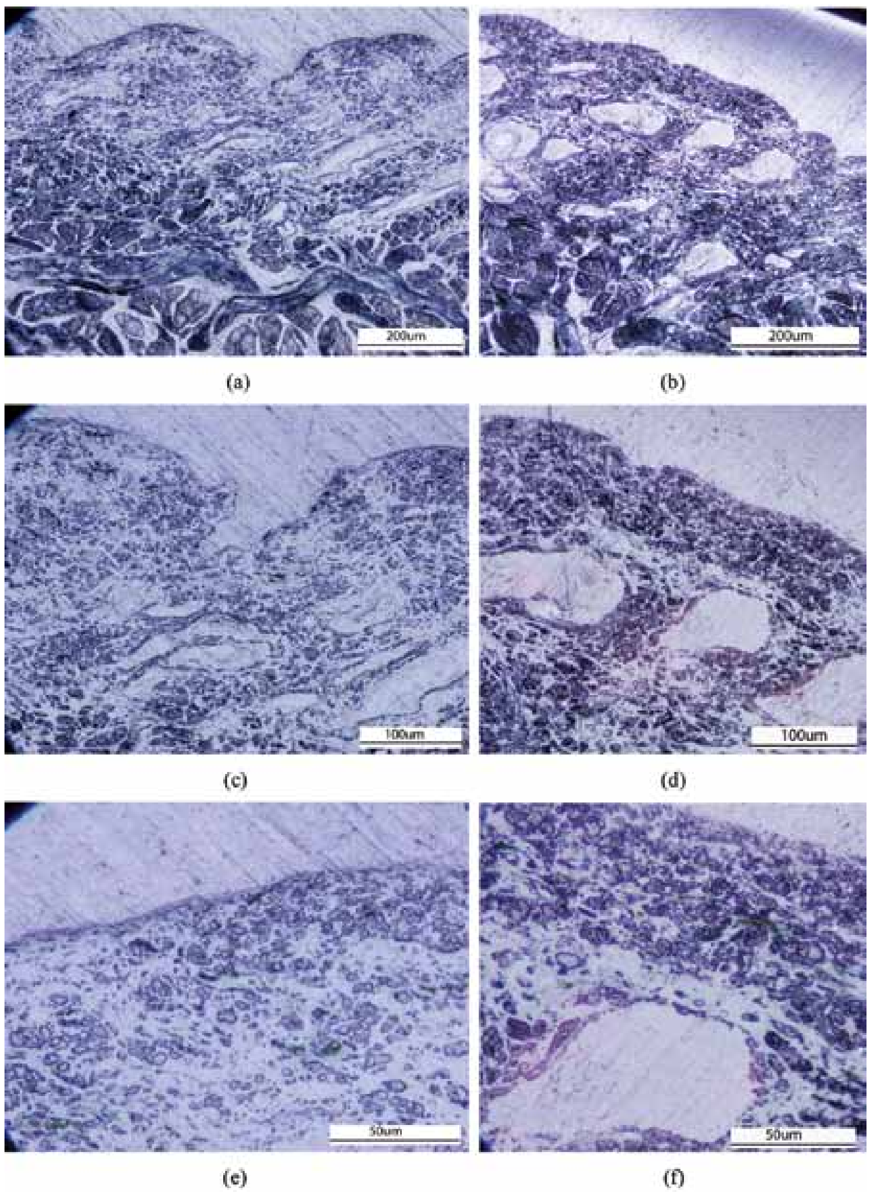


Figure 2. Vertical cross sections of grain layer.
(a, c, e — different magnification of leather sample 1;
b, d, f — different magnification of leather sample 2)

In a section of fiber bundles inclined or parallel to the image plane, the contact area of the fiber or the resin between fibers with sand paper is larger than that in a section of the fiber bundles vertical to the image plane. Consequently, the fine fiber or the tiny amount of resin between fibers are easy to drop off_when grinded or polished, which makes the inclined or parallel fiber bundle image have many fracture points and short linear pits. The fiber bundles more parallel to the observational section have longer track in the section images. Due to the coarseness of the parallel fiber surface caused by the friction, the transverse fibers would slightly stand out microscopically in the section plane, leading that the display of parallel fiber is a little indistinct. The fiber bundles run along a certain direction.

In a section of vertical fiber bundle, the vertical fiber is deeply embedded into the resin base and cannot drop off. If the fibers in the bundle are packed tightly, the bundle section would show as uniform phase (in Figure 3 a, c, e), and if the fibers are bundled loosely, many dots are shown in their image as expected that the resin between the fibers fall off (in Figure 3 b, d, f). The cross-sectional shapes of the vertical fiber bundles are approximate circle, approximate triangle, and approximate square, etc. As a result, a big fiber bundle consists of several small parts or a few fibers are aggregated to a bigger fiber bundle.

Presentation Mode of the Fine Fiber Cross Section

The fine fiber in fiber bundle can be observed more clearly where the fine fibers disperse well, such as the edge of the

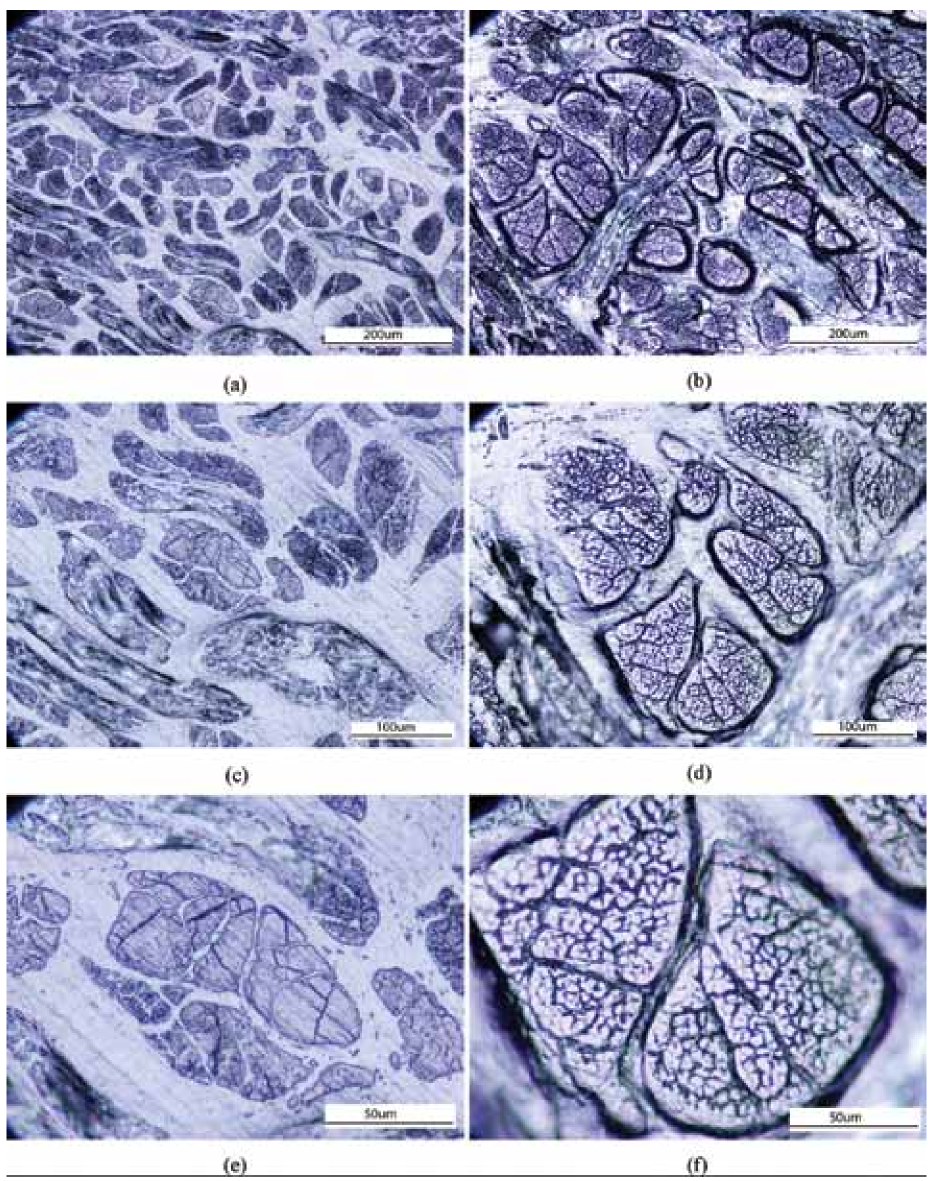


Figure 3. Vertical cross sections of reticular layer.
(a, c, e--different magnification of leather sample 1;
b, d, f--different magnification of leather sample 2)

reticular layer. In Figure 4, the sectional imagines of vertical fine fiber appear as granular, spots, and more uniform than those in the grain layer in size. Because the fiber in resin matrix has strong wear resistance and some flexibility, the fiber section would not be hurt (arrows signified in figure 4. b) where the fine scratches left by the sandpaper grinding. The section of inclined or parallel fiber shows as crooked dotted line with blank line segment, which is caused by the dropping of some part of parallel fiber (Figure 4 c).

The Boundaries of the Fiber Bundles Sections

Some fiber bundles sections appear to have either broad or narrow dark boundaries (Figure 5) depending upon the degree of cure for an epoxy resin. Due to the lower viscosity of curing agent than that of epoxy resin, the curing agent tends to be absorbed and transported more quickly in the fiber bundles than in the epoxy resin, resulting that the proportion of curing agent in epoxy resin around the fiber bundle is reduced which slow down the curing rate of the resin.

[We think that the amine groups react chemically with Cr3+ but this may not be the main reason for dark-colored complexes as seen due to the following facts:

1. Not all fiber boundaries have dark boundaries,

2. With the increasing of the curing time, some black outlines of fiber bundles disappear gradually,

3. This kind of phenomenon can also appear when the resin is unsaturated polyester with the curing system of cobalt salt and MEKPO.]

Without fully post cure, the curing degree and the mechanical strength of the resin surrounding the fiber bundle are worse than the rest of resin. Therefore, the resin in contact to the fiber bundle is easy to fall off during the process of grinding and polishing, and gives a black outline in the images. The Figure 5 (a) was acquired with common cure (not fully post cure), the black outlines exist around most fiber bundles sections. Figure 5 (b) was acquired after one week of post cure; some blank outlines where the fiber bundles are sparse disappear (such as the area marked with 1 and 4). And some blank outlines still exist where the fibers are dense (such as the marked area of 2) and the proportion of curing agent is low. After a week's post cure, the boundary of vertical fiber bundle section (such as the area marked with 4) is more easily defined, but the details of some inclined or level bundles (such as the area marked with 3) are not so clearly but fuzzy. With the increasing of the curing time, the black outlines of fiber bundles disappear gradually.

Presentation Mode of the Reticular Layer Horizontal Cross-Section

As shown in Figure 6 (a): the fiber bundles are almost parallel or inclined to the image plane. The fine fiber or resin between fibers is very easy to be scrapped off by grinding and

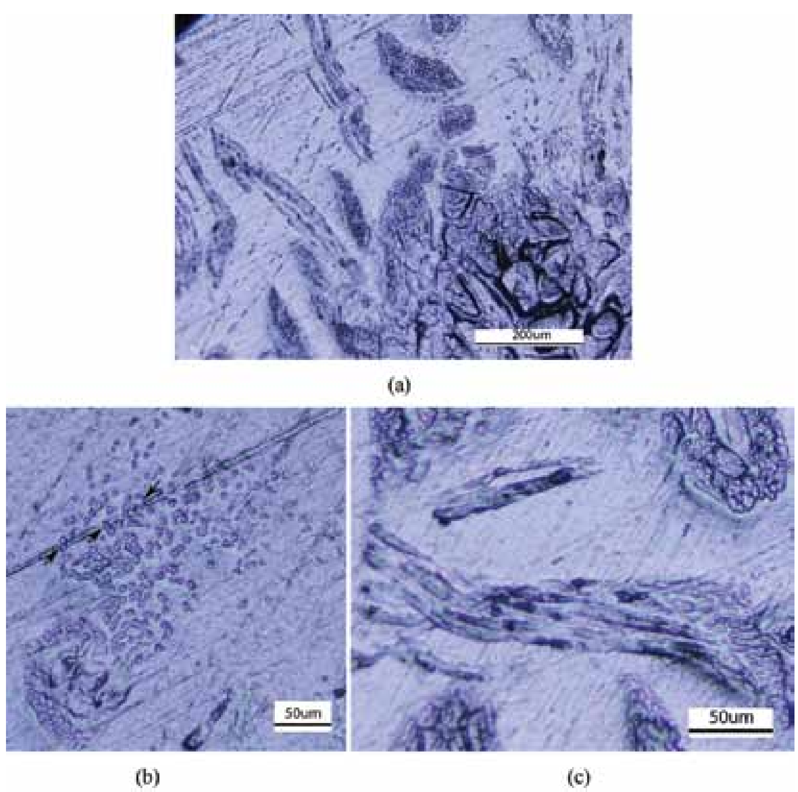


Figure 4. Cross sections of fine fibers of reticular layer.

polishing, leading to many black pits or stripes in the cross-sectional image observed by reflect light mode. Because the epoxy resin is transparent in light, the Metallographic sample was also observed by light transmission mode (Figure 6 (b)) to obtain more information; through reduce the thickness of the leather sample after embedding (less than 0.5 mm thickness) by sandpaper. Figure 6 (a) and (b) are the different images taken from the same sample, which can give a better understanding of the various orientations of the fiber bundles section. Because some fiber bundles are wavelike, a continuous fiber bundle in leather can show different orientations in a cross-sectional image, such as the parallel, perpendicular, or tilted fiber bundle (the area marked with 2).

Where the fibers on the section were scrapped off, a black deboss (pit or a hole) remained after the fibers were scrapped off. It can be observed in a reflective mode with the comparison of the smooth and some transparent view in a transmission mode (the area marked with 3). Because the dent cannot be polished well, the loss of fiber can weaken the light reflection effect, but it cannot affect the light transmission. Also the scratches, which are clearly shown as stripes in the reflective mode are almost transparent in the transmission mode (such as the places marked 4).

In addition, since the resin is transparent in the transmission mode, the trace of the fiber bundle under the current section can be found, and the continuation trend and form can be inferred. As shown in Figure 7 (a), independent fiber bundle sections 1 and 3 are jointed by the shadow 2 between the sections1 and 3, we can infer the fiber bundles sections 1 and 3 are the section of same fiber bundle. In the continuous image obtained after the section surface was grinded down about 10 um (Figure 7 (b)), we can see the shadow 2 is the real fiber section, and parts 1, 2 and 3 is a continuous fiber bundle section.

CONCLUSIONS

In this article, through a new technique for revealing the real structures of leather based on metallographic sample preparation, a series of optical microscopic images were readily obtained. The different patterns of leather sections

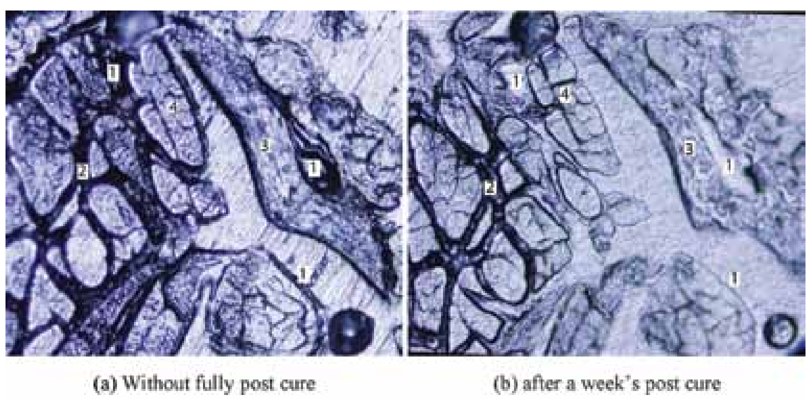


Figure 5. Comparison of the black outline with different post cure.

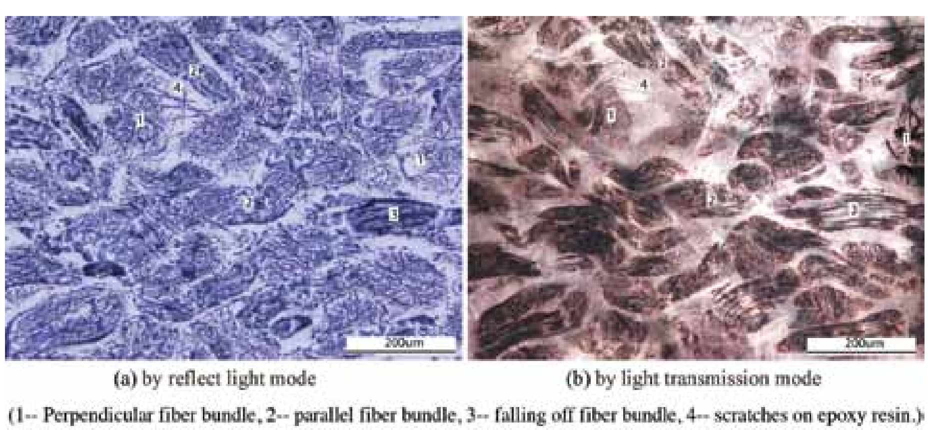


Figure 6. Horizontal cross-section of the reticular layer.
(1 — Perpendicular fiber bundle, 2 — parallel fiber bundle,
3 — falling off fiber bundle, 4 — scratches on epoxy resin.)

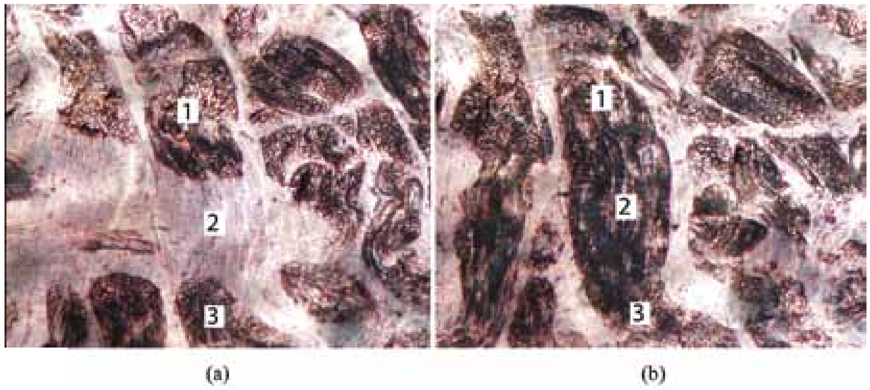


Figure 7. Trend of the fiber bundle.

were introduced, such as the vertical cross section of the grain layer and reticular layer, the fine fiber cross section and the black outlines surrounding the fiber bundles sections etc. In order to obtain more details of the fiber structure from the horizontal cross-section of the reticular layer, the metallographic samples were also observed by the light transmission mode. The difference between the patterns of fiber bundles sections obtained by the light transmission mode and by the light reflective mode was explained. With the introduction and explain of this article, the images taken from metallographic samples can be well understood, and the technique for revealing the real structures of leather based on metallographic sample preparation can be well popularized.

ACKNOWLEDGEMENTS

This study was supported by the Project of Shandong Province Higher Educational Science and Technology Program (Granted No. J11LB14).

REFERENCES

1. Pan, J.S.; Leather microstructure and the progress of leather industry. *Journal of Chinese Electron Microscopy Society* **03**, 39, 1986.
2. Rajaram, A., Sanjeevi, R. and Ramanathan, N.; The mechanical properties of collagen fibers. *JALCA* **73**, 387-393, 1978.
3. Haines, B.M., Barlow, J.R.; The anatomy of leather. *J. Mater. Sci*. **10**, 525-538, 1975.
4. Liu, C.K., Latona, N., Lee, J., L. and Cook, P.; Microscopic observations of leather looseness and its effects on mechanical properties. *JALCA* **104**, 230-236, 2009.
5. Liu, C.K., Latona, N. P. and DiMaio, G. L.; Degree of opening up of the leather structure characterization by acoustic emission. *JALCA* **96**, 367-381, 2001.
6. Yuan, J.J., Du, S.X., Yu, C.Z.; Study on relationship between histology and characterization of pig and goat leather. *Leather Science and Engineering* **21**, 19-21, 2011.
7. Tuckermann, M., Mertig, M., Pompe, W.; Stress measurements on chrome-tanned leather. *J. Mater. Sci*. **36**, 1789-1799, 2001.
8. Nishad, Fathima N., Pradeep Kumar, M., Raghava Rao, J., Nair, B.U.; A DSC investigation on the changes in pore structure of skin during leather processing. *Thermochimica Acta*. **501**, 98–102, 2010.
9. Lv, J., Yu, C.Z.; Study on histology and characterization of pigskin, sheepskin and cattle hide resultant leathers. *Leather and Chemicals* **27**, 36-39, 2010.
10. Nafstad, O., Wisløff, H. and Grønstøl, H.; Morphology of the leather defect light flecks and Spots. *Acta Vet. Scand* **42**, 107-112, 2001.
11. Zhang, H.Y., Xia, Y.M., Cheng, J.Y., Shi, L. and Li, T.D.; A novel technique for getting leather section image based on metallographic sample preparation. *JALCA* **108**, 166-170, 2013.