A Novel Technique for Getting Leather Section Image Based on Metallographic Sample Preparation

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Abstract

In this paper, the procedure of improved metallographic sample preparation used to reveal the true structure of the chrome tanned crust leather was described for the first time. E-51 epoxy resin was selected as leather embedding medium. The surface of the prepared samples was examined on an optical Microscope (Olympus BX51) using light reflection model, and the micrographs illustrating the shape and the directions of fiber bundles were obtained. This novel technique offers significant advantages over conventional one.

Resumen

En este documento, el procedimiento de preparación de una muestra metalográfica mejorada utilizada para revelar la verdadera estructura de un cuero curtido al cromo semi-terminado fue descripta por primera vez. La resina epoxi E-51 fue seleccionada como medio de embutimiento en cuero. La superficie de las muestras preparadas fue examinada en un microscopio óptico (Olympus BX51) mediante un modelo de reflexión de la luz, y las micrografías que ilustran la forma y las direcciones de los haces de fibras fueron obtenidas. Esta nueva técnica ofrece ventajas significativas sobre la técnica convencional.

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INTRODUCTION

The woven fiber structure of leather very much influences leather performance.[1] Existing leather fiber woven structure is deduced by observing and analyzing a leather section,[2-4] which can not completely reveal the true woven structure owing to its structural complex characteristic of three-dimensional (3D) form. In order to acquire the real woven structure of leather, it is imperative to build up the 3D reconstruction of fiber woven structure using a series section images with an appropriate technique to prepare and observe samples. The conventional way to get the section image of leather is through observing the leather slice, which acquired by microtomy.[5-7] 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, due to the following reasons: (1) The slices were shrunk or crimped when the embedding block was compressed by the slicer during the section process; (2) The slice were easily broken and the fibers in the slice were easily deformed or fallen out when the slice was spread out by the liquid and fished up from the liquid. As a consequence, the series section or the fiber bundle images in the sequence section images cannot be continuous. In order to solve the above problems, an improved metallographic technique was developed to prepare and examine the leather specimens in this research.

Metallographic preparation and examination is an important and straightforward method to reveal the constituents and structural features of specimens, such as metals and alloys,[8-10] in the development of modern materials science and technology. The basic steps of metallographic preparation include sample cutting, mosaicing, grinding, polishing and etching, etc.[11] The prepared samples can be readily observed in the metallographic microscope. Metallographic preparation has also been successfully applied to research on nonmetallic materials such as porous ceramics,[12] surface coating materials, [13] c-c composite,[14, 15] etc. But there is not yet a report of metallographic preparation and examination employed on the treatment and analysis of leather samples. A new metallographic technique for revealing the true leather structures has been developed by repeating some experiments hundreds of times. Through this technique, a series of optical micrographs of cross sections of chrome tanning crust leathers were readily obtained.

EXPERIMENTAL

Starting Materials

A crust leather of chrome-tanned cattle hide, obtained from the Shandong Dexin Leather Company with thickness about 1.2 mm, was chosen for this work (for simplicity, it is referred to hereafter as leather). The leather was cut into sample band (about 2mm×20mm) and dried in a drying oven at 70°C for 24 hours to remove moisture. Epoxy resin was used as the embedding medium for the leather. The grade of epoxy and its hardener were E-51 and 593#, respectively, made by Jinan TianMao Resin Company, China. This epoxy resin was suitable for the embedding process due to its lower viscosity, better mechanical and adhesive properties, less contractibility than other polyester resins. The mass ratio of epoxy / hardener was 4/1 for the preparation of all the samples.

Specimen Preparation

The entire process was designed to produce a scratch free surface with real structure by embedding, mounting and a series of successively grinding/polishing. Each stage of specimen preparation must be carefully performed. Failure to be careful in any step would result in an unsatisfactory sample.

1. Mold Preparation

Small samples are generally mounted in a hard polymer block with the diameter of 1 to 1.5 inch for convenience in handling and to protect the edges of the specimens being prepared. A mold can be prepared by the following steps: Prepare a glass plate (about 8mm×8mm) coated with demoulding agent and a paperboard tube (diameter Ø35mm, length 30mm). Second, put the paperboard tube on the glass plate, and then use plasticine to bond them together and to seal the gap between them.

2. Embedding

The leather samples are soft and composed of a dense plexus of fibrous bundles, knit together and interwoven in every direction, and the interspaces between fibers are very small. It is difficult to be stationary if the leather is mounted directly without pretreatment when impregnated by the resin. Therefore, rigidity is required, that is, an embedding progress is needed as follows.

The embedding method consists of vacuum impregnation with a slow curing epoxy (the epoxy resin- hardener ratio of 5 to 1) followed by both room temperature and pressure curing. This was accomplished by the following steps: put a normal paper cup filled with 2 mm thick epoxy into a vacuum chamber, then kept a leather sample flatting on the surface of the epoxy and vacuumized the chamber simultaneously. After the system reached to a pressure of -0.9 MPa and subsequently was held for 1 min, the cup was taken out to cure at room temperature and atmospheric pressure. Finally cut off the leather band from the resin cylinder and exposed the examination surface by cutting and grinding on 800 grit SiC paper.

3. Mounting

Small samples can be difficult to hold safely during grinding and polishing processes, and difficult to offer a flat surface for observation. They are therefore mounted inside a polymer block. The specimens can be mounted in any of the common metallographic mounting materials, such as Bakelite, phenolic, and epoxy. However only epoxy can be used in cold mounting, which is suitable to the leather character. The sample after embedding was placed in the center of the mold with the examination surface of the leather glued to the glass by fast-setting resin (502), then the epoxy resin (mixture) was poured in and allowed to cure at room conditions for about 2 hours. Postcure was followed at 60ºC for 2 hours in order to get a complete cure resin. Subsequently, the glass and paperboard tube of the mold was removed and the mounted specimen (figure 1, about Ø35mm diameter, 20mm height) was obtained.

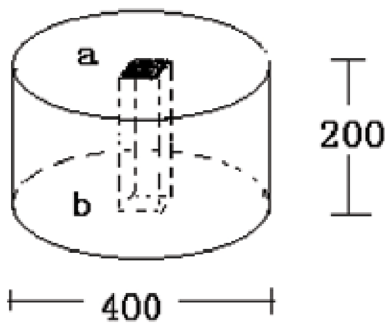

Figure 1. Sketch of mounted specimen.

4. Coarse Grinding

The purpose of coarse grinding stage is to generate the initial flat surface necessary for the subsequent fine grinding and polishing steps. As a result of sectioning and grinding, the material may be worked to a considerable depth where the material structures are not distorted by the former processes. Coarse grinding can be accomplished either wet or dry using 80 to 180 grit electrically powered disks or belts, but care must be taken to avoid significant heating of the sample. The final objective is to obtain a flat surface free from all previous tool marks.

5. Fine Grinding

Fine grinding of samples are closely allied with the coarse grinding which precedes it. The purpose is to carefully move from one stage to the next where the abrasives become finer at each successive stage. Because the leather and epoxy resin are soft and easy to grind away, manual wet grinding is used for leather samples which is generally applied in fine grinding to avoid possible side affects due to heating such as tempering, transformation, aging, incipient melting, etc. Wet grinding also provides a flushing action for loose particles and keeps sharp edges of the grinding medium exposed at all times. Successive stages are grinding on 240, 320, 600, 1000, 1500, and 2000 grit SiC papers.

It is important that abrasive is not carried from one grinding stage to another. Therefore, you must wash both the specimens and your hands between each stage. When the specimens are ground, they are rubbed forward in one direction until the surface is completely ground (that is, until the scratches are uniform in size and parallel to each other in any one grinding stage), and they should be rotated by 90º between stages. Failure to follow this basic rule can result in transferring abrasive particles between stages and will cause time-consuming, frustrating problems in removing unwanted scratches.

For leather, further grinding for a short time is advisable after this condition is reached to remove any sub-surface deformation produced in previous operations.

6. Polishing

The purpose of polishing is to have a mirror-like surface free of scratches. Polishing is carried out on a napped cloth with 1-micron suspended aluminum oxide particle (suspended in water) covered a rotating wheel at a speed of 100 - 120 rpm. The specimens must be washed and dried before polishing steps, because even hard dust particles in the air, which settles on the polishing cloth, can cause unwanted scratching of the specimen. During the polishing, the specimen should be held firmly in contact with the polishing wheel, and should be rotated or moved around the wheel to give an even polish. Undue pressure should be avoided. Figure 2 is the micrographs with different polishing time. Figure2 (b) and (c), both show the clear boundaries of leather fiber bundles, but some areas (where arrow indicates) in figure 2 (c) is not so clear as that in figure 2 (b) due to the missing of some boundary details by excessively polishing. So the proper polishing time is about 15 seconds.

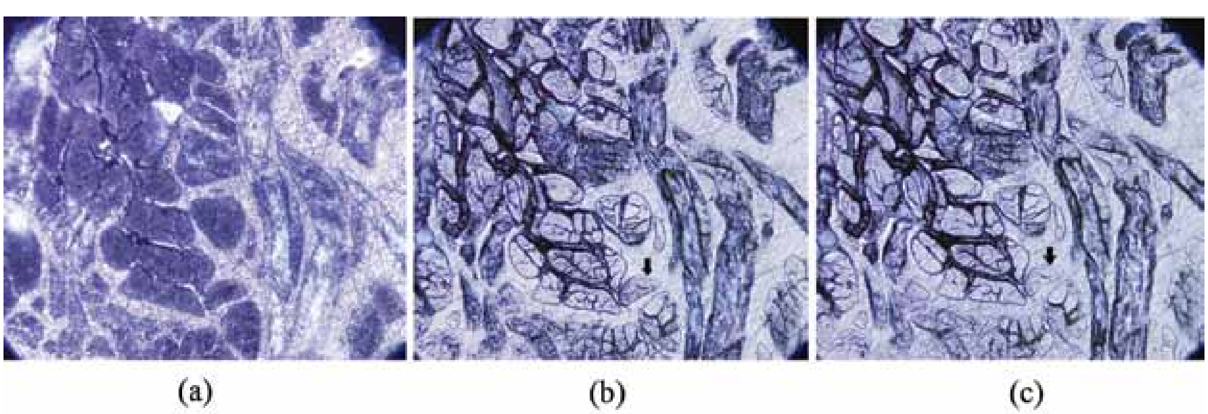

Figure 2. The optical micrographs of the leather specimen polished for (a) 0s, (b) 15s, and (c) 30s.

Microscopic Examination
Detailed viewing of the mirror-like surface (leather section) is done on an optical Microscope (Olympus BX51) using light reflection model. The microscope has a system of lenses (objectives and eyepiece) so that different magnifications (100× to 1000×) can be achieved.

RESULTS AND DISCUSSION

Figure 3 and 4 are the microscopic images of Cross section of chrome tanning crust leather, showing different region of the sample at different magnifications. As seen in both figure 3 and 4, there are no air bubbles, holes or significant scratches in section graphs. The leather organization structure is intact, and the shape of fiber bundle is clear and easy to be identified. The papillary layer and the reticular layer have a clear boundary, either of which has its own characteristic. The fiber bundles of papillary layer are fine, knitting closely, while the fiber bundles of reticular layer are thick, dispersed uniformly. The vertical and horizontal fibers all keep natural state, no deformation and no overlap.

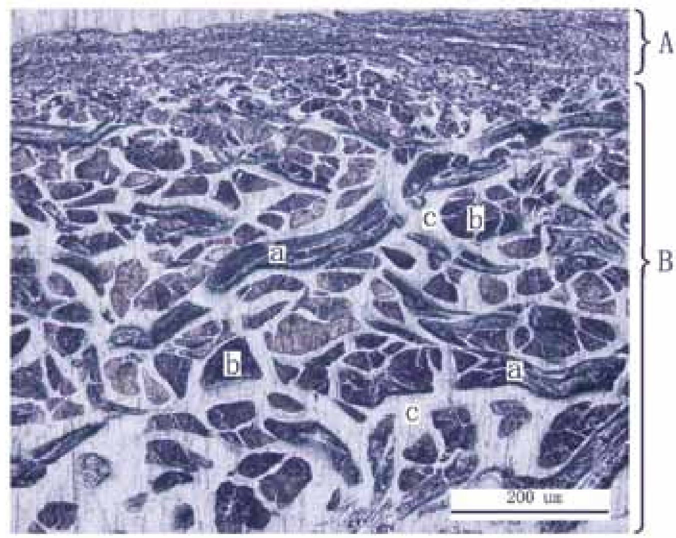

Figure 3. The microscopic images of cross section of chrome tanned crust leather sample consisting of two layers, A — papillary dermis, B — reticular dermis, (a — fiber bundles which parallel to the section, b — fiber bundles which perpendicular to the section, c — epoxy resin.)

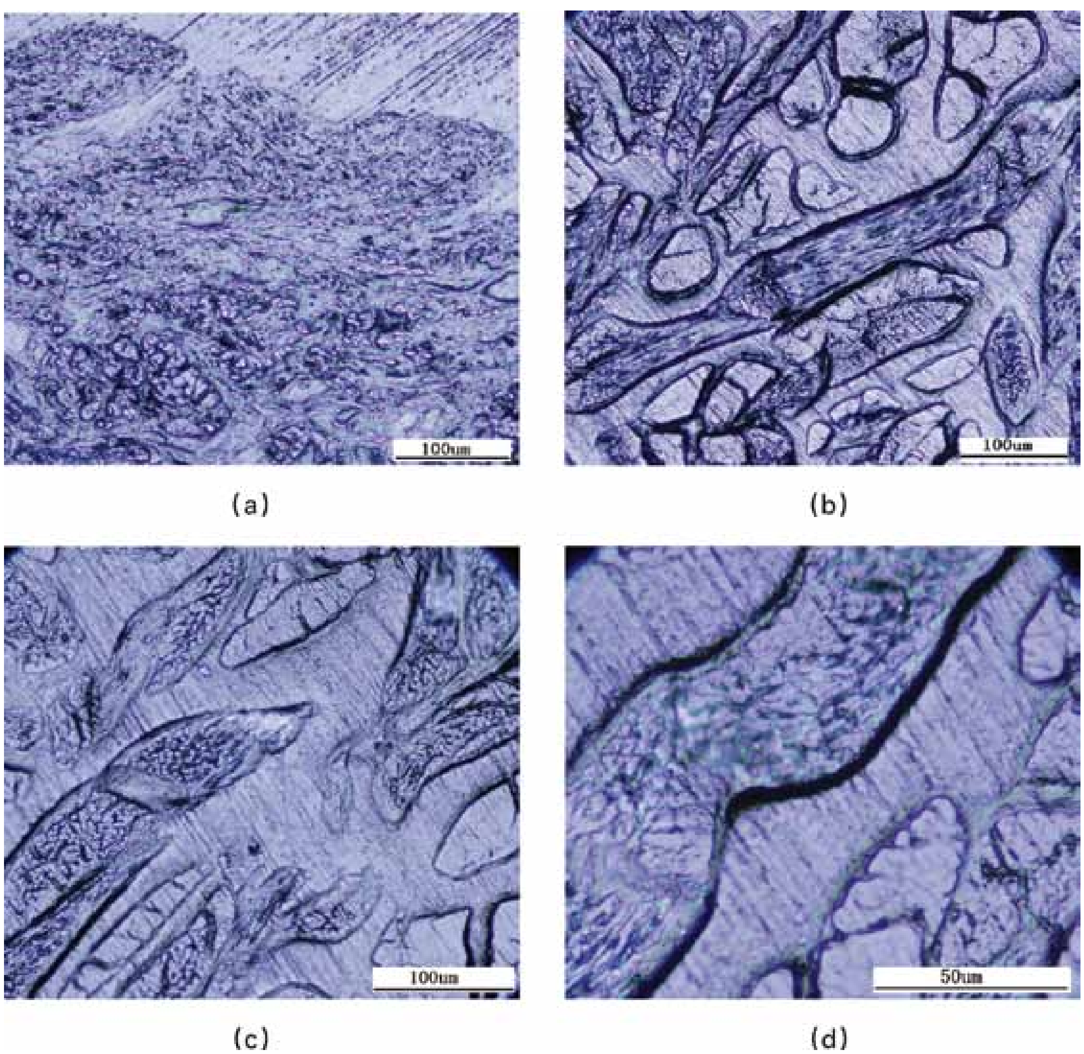

Figure 4. The microscopic images of (a) papillary dermis and (b), (c) and (d) reticular dermis at different magnifications.

Those obvious advantages of preparing leather sample for microscopic observation can be attributed to: (1) E-51 Epoxy resin whose viscosity and contractibility is low has excellent permeability and adhesive property for leather. (2)The vacuum embedding to little leather makes the resin fill up the interspaces between the fibers completely and avoid forming air bubbles. Considering the little mount of the resin for embedding, the quantity of heat released from the resin curing is little and dissipates soon so that to avoid the deformation and damage to the leather fibers. (3) Grinding and polishing are operations on the section of massive resin matrix including embedded leather after mounted. With the support of massive resin matrix, those operations cannot cause the fiber to deform, move and fall off.

From figure 4 (b) (c) and (d) we can see: the outer margins of fiber bundles present as deep color lines or grooves, which make the fiber bundle boundary apparent. The fiber bundles perpendicular, inclined, and even parallel to the observational section, have their own characteristics respectively in the images and are easy to be distinguished. The section of vertical fiber bundles seems to be less fractured than the others. In a section of inclined fiber bundle, the area of the resin between fibers grinded or polished by the sandpapers or the napped cloth is larger than it in a section of the vertical fiber bundles, so that the tiny resin between fibers is easy to drop off, which makes the inclined fiber bundle image have many fracture points and short linear pits. Consequently, the vertical fiber bundles and inclined ones are easier to be distinguished. The fiber bundles more parallel to the observational section will have longer track in the section image.

Conclusions and Application Prospects

In this article, a new technique for revealing the real structures of leather based on metallographic sample preparation was emphasized. Through this technique, a series of optical microscopic images with clear shape of the fiber bundles keeping natural state, without deformation or overlap, were simply obtained; the fiber bundles which were extended in different directions can be distinguished easily. This technique can be combined with layer-by-layer sanding method, to acquire sequence images with random interlamellar spacing, sequentially built base for the three-dimensional reconstruction of weaving structure of leather fibers, and provide an opportunity to further study the relationship between leather structure and leather performance.

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