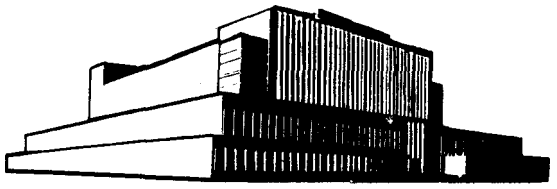

U.S. Forest Service Research Note

UNITED STATES DEPARTMENT OF AGRICULTURE • FOREST SERVICE • FOREST PRODUCTS LABORATORY , MADISON, WIS

In Cooperation with the University of Wisconsin

METHODS USED AT THE FOREST PRODUCTS LABORATORY FOR PREPARING CROSS SECTIONS OF PAPER AND PAPERBOARD

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METHODS USED AT THE FOREST PRODUCTS LABORATORY FOR PREPARING CROSS SECTIONS OF PAPER AND PAPERBOARD

By

RICHARD A. HORN, Forest Products Technologist
and
S. C. ELLICKSON, Physical Science Technician

Forest Products Laboratory, [1] Forest Service
U.S. Department of Agriculture

Summary

Methods for preparing cross sections of paper are discussed and related to the type of study each would be appropriate for. Nonembedding methods as well as embedding techniques are described.

Introduction

Knowledge of the structure of paper and paperboard is basic to understanding their behavior, improving their quality, and broadening their usefulness. Structure is considered here to be the arrangement and appearance of the fibers and the distribution of nonfibrous materials that may be present.

Microscopy is the only direct means for studying structure. Available microscopical techniques are optical, including fluorescence and ultraviolet, electron, and X-ray.

Areas of fiber-to-fiber bonds, where fibers cross in paper surfaces, can be observed by polarized vertical illumination in the visual spectrum. This was demonstrated by Page (9)[2] and exploited quantitatively by him and his coworkers (10). Other research on the orientation of fibers in paper was done by Danielsen and Steenberg (2), Forgacs (3), and Prusas (11). The surface and internal distribution of starch and rosin in papers was shown by Lee (8). The internal distribuion of chemicals in linerboards is under study at the Forest Products Laboratory in cooperation with the U.S. Air Force.

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[1] Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

[2] Underlined numbers in parentheses refer to Literature Cited at the end of this report.

The study of internal structure of paper and paperboard requires the preparation of thin sections of samples. These sections may be parallel with the machine direction, across the machine direction, or parallel to the plane of the paper or board sample. Methods for preparation range from simple freehand cutting of unembedded specimens to tedious embedding and cutting with a microtome. Various methods for sectioning paper have been described by Graff and Schlosser (5) and Graff (4).

When nonfibrous components are to be identified and their distribution studied, embedding the specimen in one of the waxes or resins used for this purpose is undesirable because usually the component will be obscured or there will be interference with staining reactions. Although some embeddents can be removed with a solvent, this is, in general, undesirable because nonfibrous components may be affected with respect to location or response to stain.

Embedding can be avoided by using the technique of Banks and Evans in which the specimen is placed between two sheets of polyethylene softened at 110° C. and sectioned when cool by a microtome (1). The freezing microtome has been recommended by Jayme and Harders-Steinhauser for cutting sections (6). Embedding in ice avoids the tedious procedures of using waxes or resins andtheuse of solvents for their removal. If, however, any distortion of the section must be avoided, this technique is not suitable. Nonembedded sections are of especial interest to the Forest Products Laboratory in connection with studies of the identification and distribution of nonfibrous components of experimental papers and paperboards.

Freehand, Nonembedding Methods

In current investigations at the Forest Products Laboratory of the identification and distribution of chemicals in linerboards, it was found that satisfactory sections could be obtained freehand with a single-edge safety razor blade. The specimen used in this procedure is a strip about 3/8 inch wide and of convenient length cut parallel with the machine direction of the linerboard (fig. 1). The strip is held firmly, wire side uppermost, on a smooth-surfaced paperboard, and one edge parallel with the machine direction is beveled at an angle of about 45°. Sections across the machine direction are cut, using a slight slicing action with the blade and using the forefinger as a guide. Only a little practice is required to produce sections thin enough for examinationwith transmitted light. When the section is viewed, the shorter surface is the wire side of the board.[3] Papers as thin as 0.006 inch can be sectioned with this technique.

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[3] Polarized light inconjunctionwith the first order red plate is often superior to normal light for examination of these sections with transmitted light. Reflected light can be used also.

When paper samples less than 0.006 inch in thickness are to be sectioned across the machine direction, a specimen 1/2 inch wide and about 2 inches long is cut parallel with the machine direction. This piece is folded lengthwise and the cut is made with a razor blade across the machine direction (fig. 2). Sections almost as thin as microns have been prepared in this way. The V-form maintains the desired surface in proper position, and either surface is easily identifiable.

Microtome Nonembedding Method

When nonembedded sections are to be thinner than can be cut freehand, a microtome is used. Because paper and paperboards are too flexible for sectioning in the microtome clamp, a support of sufficient rigidity must be provided.

A variation of the Banks and Evans sandwich method (1) is used at the Forest Products Laboratory for this purpose. A 1/4- by 3/4-inch strip is cut from the stock sample, and two 3/4- by 1-1/2 inch pieces are cut from cellulose acetate film 0.02-inch thick. The pieces of film are placed on a smooth surface and treated with acetone to soften them. The acetone is applied to the center of the film with a medicine dropper, using 2 to 3 drops. This permits the acetone to flow evenly over the surface. The softening action of the acetone is allowed to continue for approximately one minute. The paper specimen is then sandwiched between these softened surfaces and the sandwich placed in a press consisting of two pieces of cork glued on the inside of a burette clamp (fig. 3a). Enough pressure is applied so that the softened film surfaces will flow. This results in a homogeneous support for the paper sample.

The sandwich is allowed to dry 15 minutes in this press under room conditions. It is then trimmed with scissors or a razor blade, leaving a 3-millimeter margin of plastic film on one edge and a shorter margin on the other edge as shown in figure 3b. The specimen is placed in the microtome clamp so that the 3-millimeter edge is toward the knife and a cut of approximately 2 millimeters is made into this edge to produce a section. This section of the sandwich is held against the microtome knife by placing a dissecting needle horizontally across the section as it is being cut (fig. 3c). Because of the hygroscopic nature of the sections, there is a tendency for them to curl and often split. To prevent this from occurring, a section is placed on a glass slide, covered with a cover slip on which a 50-gram weight is placed, and kept in a desiccator.

Microtome Embedding Methods

Occasions arise when it is necessary to obtain information on the sample in its entirety without any distortion of the section. Paper sections as thin as 5 to 10 microns can be cut, but the sample must be embedded in some material; otherwise, the thin sections would disintegrate.

One of the most common materials used for embedding is paraffin. The paraffin embedding method is easily applied to paper and, with practice, it is relatively simple to prepare satisfactory sections. The specimen used for this procedure is a small strip about 1/8 inch wide and 1/2 inch long, cut parallel with or across the machine direction of the paper. The specimen is placed in a container of melted paraffin and enough time, depending upon thickness of the paper, is allowed for complete infiltration. Following infiltration, the contents are poured into suitable trays, the specimens arranged in proper order, and the trays immediately placed in ice water for rapid cooling. After the wax has hardened, the wax block is trimmed and fused to a wooden block for sectioning in the microtome. The cut sections are affixed to a glass slide with Haupts' adhesive (7) and washed with xylene to remove the paraffin followed by two or three washings with ethanol to remove the xylene.

Another technique for preparing thin sections (5 to 10 microns) using an epoxy resin[4] has been found to be satisfactory. The specimen used in this procedure is identical to that used in the paraffin technique. The resin is prepared according to instructions given by the manufacturer, except that the best results are obtained using 3 milliliters of the Nysem plasticizer in the resin mixture. This mixture is poured into type 00 capsules and, by using a needle, the specimen is positioned in the center of the capsule. These capsules are then cured for 16 to 20 hours at 48° C. A convenient receptacle for the capsules can be made by drilling holes of proper diameter and depth in a block of wood. The capsules are dissolved away from the cured blocks and then trimmed and sectioned in the usual manner.

This technique is used to study the penetration of inks or colored coatings in paper. It is not suitable when nonfibrous components are to be revealed or identified owing to interference of the embeddent. It may also interfere when ultraviolet spectrometry is to be used for identification.

Embedding methods have the disadvantage of being tedious and requiring a considerable amount of time, apparatus, and technical skill in operation. Because of these disadvantages, it is advisable first to determine whether the analysis of a sample would require such lengthy procedures. For the most part, the freehand and nonembedding techniques are simple, require little technical skill and apparatus, and usually will give the information desired.

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[4] Nysem Epoxy Resin A, distributed by R. P. Cargille Laboratories, Inc., New York.

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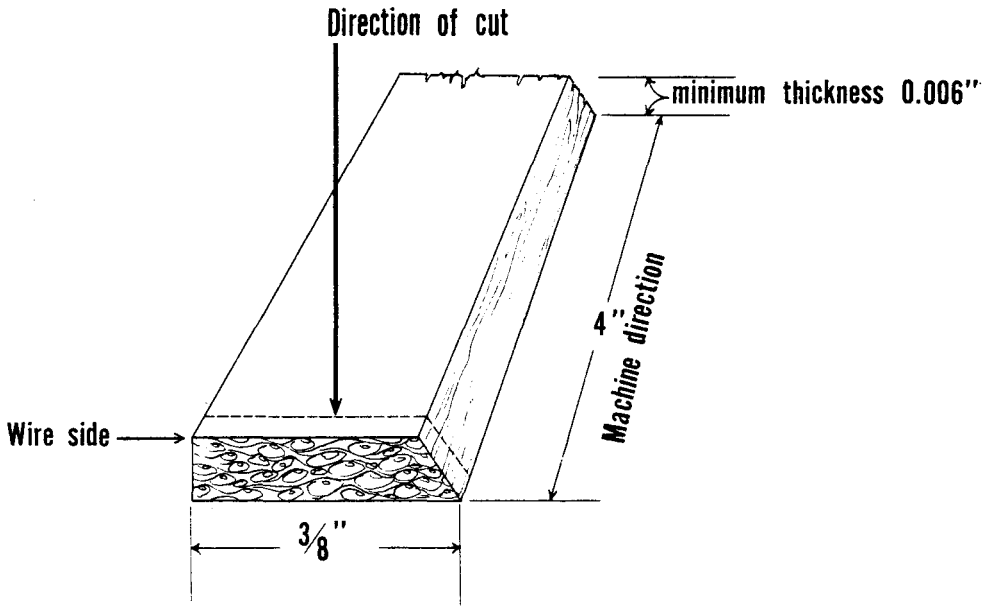


Figure 1.--Specimen of linerboard for cutting sections across machine direction freehand with single-edge safety razor blade.

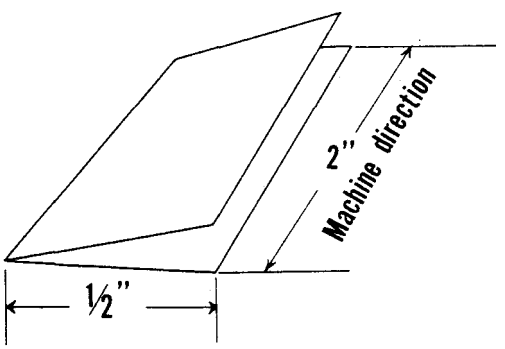
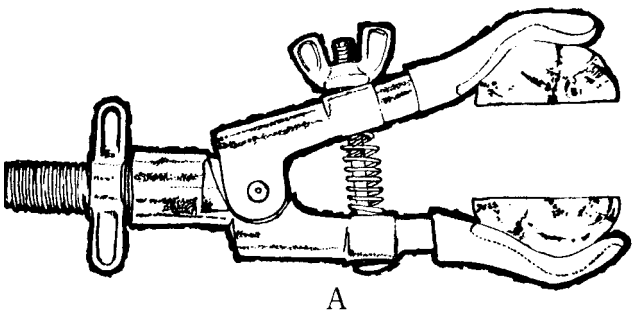


Figure 2.--V-form of specimen for cutting sections freehand from papers less than 0.006 inch thick.



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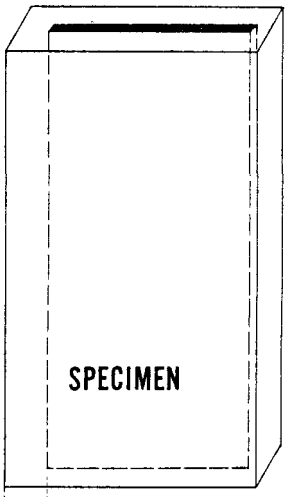

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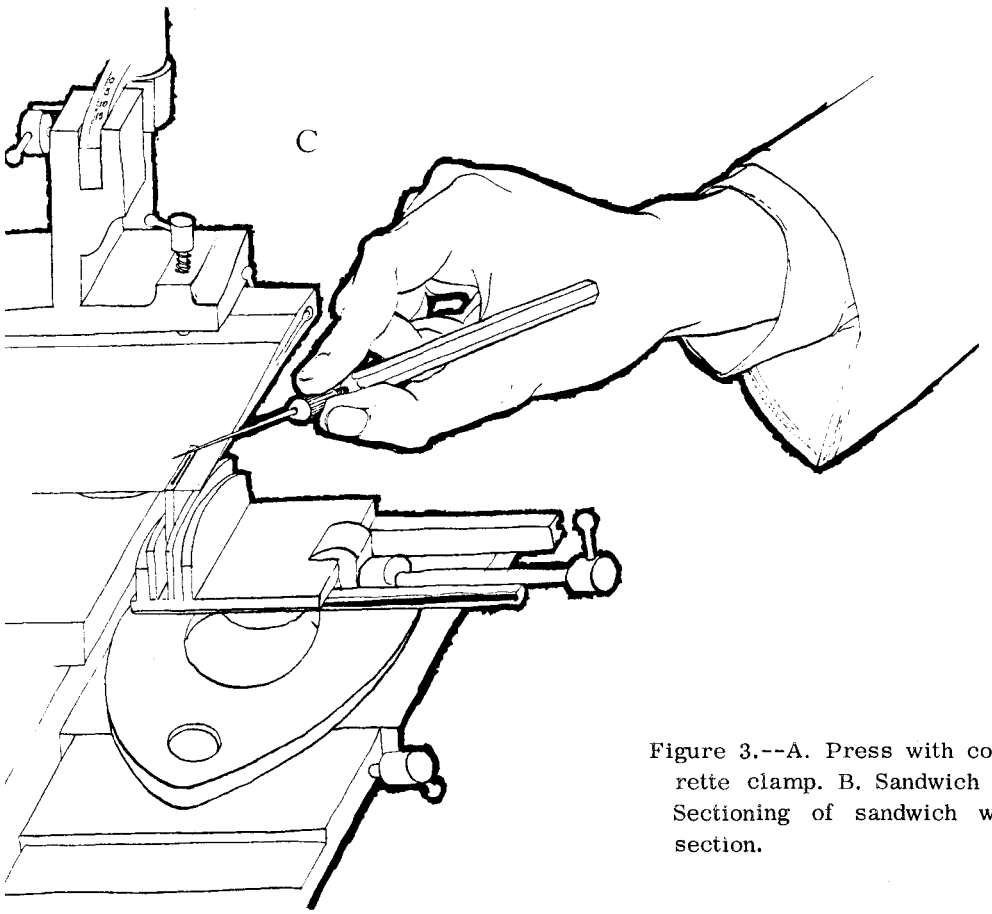

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Figure 3.--A. Press with cork glued inside burette clamp. B. Sandwich after trimming. C. Sectioning of sandwich with needle against section.

SUBJECT LISTS OF PUBLICATIONS ISSUED BY THE FOREST PRODUCTS LABORATORY

The following are obtainable free upon request from the Director, Forest Products Laboratory, Madison, Wisconsin 53705.

List of publications on
Box and Crate Construction and Packaging Data

List of publications on
Chemistry of Wood and Derived Products

List of publications on
Drying of Lumber

List of publications on
Fire Performance

List of publications on
Fungus Defects in Forest Products and Decay in Trees

List of publications on
Glue, Glued Products, and Veneer

List of publications on
Growth, Structure, and Identification of Wood

List of publications on
Logging, Milling, and Utilization of Timber Products

List of publications on
Mechanical Properties and Structural Uses of Wood and Wood Products

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List of publications on
Structural Sandwich, Plastic Laminates, and Wood-Base Aircraft Components

List of publications on
Thermal Properties of Wood

List of publications on
Wood Finishing

List of publications on
Wood Preservation

Partial list of publications for
Architects, Builders, Engineers and Retail Lumbermen

Partial list of publications for
Furniture Manufacturers, Woodworkers, and Teachers of Woodshop Practice

Note: Since Forest Products Laboratory publications are so varied in subject no single list is issued. Instead a list is made for each Laboratory division. Twice a year, December 31 and June 30, a list is made showing new reports for the previous 6 months. This is the only item sent regularly to the Laboratory's mailing list. Anyone who has asked for and received the proper subject lists and who has had his name placed on the mailing list can keep up to date on Forest Products Laboratory publications. Each subject list carries a listing of all other subject lists.