Mechanical Characterization of Wood-Adhesive Interphase with an Improved Nanoindentation Technique

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1. Introduction

- Adhesive penetration into wood structure is believed to be vital to the durability of wood-adhesive bondlines (Frihart, 2009).

- Flowing into the micron-scale cavities of wood

Fluorescence microscope and CLSM can directly show the distribution of adhesive in wood at micron scale.

- Infiltrating into the cell walls.

SEM-EDAX, UV microscopy, Thermal AFM et al
1. Introduction

◆ Understanding how infiltrated adhesives affect wood cell wall properties is critical to understanding how to make durable wood-adhesive bondlines.

◆ Nanoindentation was recently used to characterize wood-adhesive bondlines, which is based on the reasonable assumption that the infiltration of adhesives into wood cell walls might change their mechanical properties.

Nanoindentation testing

Scan Size: 4 µm * 4 µm
1. Introduction (cont)

◆ Problem 1: sample preparation

- Spurr resin
- Embeddment
- Polished with Ultramicrotome

- Long time (2-3 day);
- Cell wall may be chemically modified
- Embedment medium might interfere with the adhesive distribution in the bonding area
Problem 2: suitability of Nanoindentation

Oliver-Pharr method (standard method):

Assumptions:
• homogeneous half spaces;
• isotropic
• rigidly supported in the testing machine

\[
E_r = \frac{\sqrt{\pi}}{2 * C_p} \frac{1}{\sqrt{A_c}}
\]

\[
H = \frac{P_{\text{max}}}{A_c}
\]

\[
C_p = C_t - C_m \quad \text{(constant)}
\]

• \(C_p\): actual unloading compliance
• \(A_c\): projected area of indents, obtained by calculation.
1. Introduction (cont)

Assumptions might be violated in the nanoindentation testing of wood cell wall or adhesive bonding

**Improved nanoindentation test** (Jakes et al., 2008; 2009)

\[ C_p = C_t - C_m - C_s \]

- \( C_p \): measured with AFM
- \( A_c \): measured with AFM

- \( C_s \): Structural compliance induced by the heterogeneity, free edge, cracks, pores of samples
2. Materials and Methods

- Southern pine veneers with a thickness about 5 mm were bonded together with PF, UF, epoxy and EPI adhesives with the bonding parameters shown in Table 1.

<table>
<thead>
<tr>
<th></th>
<th>Temperature (°C)</th>
<th>Time (Min)</th>
<th>Pressure (MPa)</th>
<th>Application Rate (g/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PF</td>
<td>158</td>
<td>6</td>
<td>1.2</td>
<td>80</td>
</tr>
<tr>
<td>UF</td>
<td>125</td>
<td>5</td>
<td>1.4</td>
<td>150</td>
</tr>
<tr>
<td>Epoxy</td>
<td>Room temperature</td>
<td>180</td>
<td>0.86</td>
<td>174</td>
</tr>
<tr>
<td>EPI</td>
<td>Room temperature</td>
<td>Overnight</td>
<td>1.4</td>
<td>180</td>
</tr>
</tbody>
</table>
Sample preparation

- **Unembedment method** (Jakes et al, J Mater Res, 2008)

  ![Diagram of sample preparation]

  - Polished with Ultramicrotome
  - 1 h
  - Cell wall remains natural state
Equipment

Triboindenter (Hysirton, USA)
Cs calculated from SYS plots

![Graph of Wood cell wall](image)

Typical multiload curves

SYS plots

\[ C_t L_{\text{max}}^{1/2} = (C_m + C_s) L_{\text{max}}^{1/2} + J_{0}^{1/2} \]

Dependent variable

Variable
Projected area measured with AFM

Wood cell wall

PF

UF

EPI

Epoxy

Scan Size: 4 µm * 4 µm
3. Results and discussion

Typical load-depth curves of multiload indents on the four adhesives
Comparison between standard method and corrected method in MOE and hardness
3. Results and discussion (cont)

The elastic modulus and hardness of the four wood adhesives
The effect of distance to PF adhesive line on the elastic modulus and hardness variation of wood cell wall.
UF bonding

Bonding line

Locations in the bonding line

Elastic modulus (GPa)

Hardness (GPa)

Elastic modulus

Hardness
EPI bonding
4. Conclusion

- Of the wood adhesives tested, PF has the highest elastic modulus and hardness, followed by UF, epoxy and EPI in turn.

- Wood cell walls near UF and PF bondlines had increased hardness, but the elastic modulus was not modified.

- Mechanical properties of wood cell walls near epoxy and EPI bondlines were not modified.

- The durability and strength of wood-PF bondlines is likely because of the high mechanical properties of the PF itself and its ability to infiltrate and strengthen wood cell walls near the bondline.
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Any question?