XMT Imaging and Digital Volume Correlation for Characterization of Micromechanical Performance in Wood-Plastic Composites

Lech Muszyński
Brian K. Bay
John Simonsen
Yu Geng
Wood-Plastic Composites (WPCs)

Heterogeneous dispersed particulate composites comprised of 3 phases:

- **Particles**: Wood or other natural fibers
- **Matrix**: Thermoplastics
  - Polystyrene (PS)
  - Polyethylene (PE, HDPE)
  - Polypropylene (PP)
  - Polyvinyl chloride (PVC)
- **Additives…**

[Composites.wsu.edu/ navy/Navy1/materials.html](composites.wsu.edu/ navy/Navy1/materials.html)
Additives (up to 10%)

- Lubricants/Process aids
- Colorants
- Coupling agents
- Preservatives
- Antimicrobials
- Antioxidants
- Flame retardants
- Light (UV) stabilizers
Issues...

- Durability
- Significant creep
- Thermo-expansion
- Weight/Strength
Wood-Plastic Composites (WPCs)

- Mechanical and physical properties of WPC’s may be engineered by changing phase characteristics, proportions and orientation, processing parameters, and internal bonding.

- **Limitations**: composite design is limited by the naturally variable wood properties and the limited selection of thermoplastics that may be used with wood (melting temperature <200° C)
Focus on the Internal Bond

- Challenge: Bonding two dissimilar materials
  - Hydrophilic wood
  - Hydrophobic thermoplastics
- Facilitated by coupling agents
Motivation

- Durability and mechanical performance of WPC’s are decided on the µ-mechanical level
- Traditional testing methods offer indirect and limited insight to µ-mechanical performance, µ-damage accumulation and governing failure mechanisms
- Any significant progress in this field depends on better understanding of the composite performance and internal bond durability on the µ-mechanical level, and reliable modeling based on that understanding
How much can flexural tests reveal about internal bonding?
PROBLEM STATEMENT

- Can the external measures of the internal bond performance be correlated to micro-mechanical characteristics of the composite materials (WPC’s)?

- Can WPC material degradation due to long term environmental exposure be simulated by mechanical loading?
OBJECTIVES

To develop experimental procedures for multi-scale evaluation of micro-mechanical performance, governing failure mechanisms and micro-damage accumulation in WPC’s.
OBJECTIVES

The specific objectives are to investigate:

- Statistical characterization of the local deformation and internal damage accumulation induced by degrading conditions;

- Correlations between the internal damage and the storage modulus;

- Correlations between the internal damage inflicted by mechanical loading and cyclic environmental exposure regimes: soak-dry and freeze-thaw.
Procedure: Multi-scale analysis

- Macro level: elastic modulus, static strength, storage modulus after degrading treatment

- Optical DIC analysis to identify strain concentrations on the specimen surfaces

- Digital Volume Correlation based on low resolution CT scans (20 µm/pixel) to identify internal strain concentrations (AOI for hi-res CT)

- High Resolution CT scans (3 µm/pixel) on the AOI’s identified in the previous steps to reveal and characterize internal damage concentrations
Damage characterization

Damage is defined as a degradation in microstructure due to an external or internal influence.

- mechanical stress
- moisture/temperature cycling
- chemical changes

Define damage variable, $D$, such that $E = E_0(1 - D)$
Nondestructive X-Ray Microtomography

Nondestructive measurement allows evaluation of the three-dimensional internal structure before and after the degrading treatments.
X-ray Microtomography: Measurement principles

- 3D maps of x-ray absorption reconstructed from projection images
- High resolution through high performance x-ray source and detector
Nondestructive 3-D X-ray microtomography

Is it good for more than just cool pictures?

oak 2 mm  oak 2 mm
LVL (transverse plane)  2 mm
plywood, birch  2 mm
OSB  5 mm

Oak vessels  top
front
right side
back

WPC: Wood/PP  1 mm
FRP: E-glass/urethane  2 mm
FRP: E-glass/epoxy  2 mm

Who’s next?

Wood/PP  1 mm
GF/Urethane  2 mm
GF/Epoxy  2 mm

2005 SWST/FPS, Quebec City, Canada
Nondestructive 3-D X-ray microtomography

Is it good for more than just cool pictures?

- Digital tools are available to identify visible features and different material phases (solid phases, voids etc.), quantify connected pore structure, and visualize complex microstructure.

- Internal strains can be evaluated (Digital Volume Correlation).
Digital Image Correlation
Digital Volume Correlation
Types of Image Correlation

- In-plane deformations from single-camera surface images
- Out-of-plane deformations from multiple-camera surface images
- Volumetric deformations from tomographic data sets (Digital Volume Correlation)
  - A 3D extension of DIC
  - Applicable to materials with inherent texture
  - Porous materials, composites, large-scale microarchitecture

2005 SWST/FPS, Quebec City, Canada
Volumetric Strain Measurement - Concept

Strain is quantified using correlation methods that compare loaded and unloaded data volumes.

1. Marker Tracking in 2D
2. Speckle Metrology in 2D
3. Texture Correlation in 2D
4. Texture Correlation in 3D
Integrated Testing and Imaging

Detector   Sample Stage   X-ray Source

2005 SWST/FPS, Quebec City, Canada
Hardware Details

- **The x-ray source:**
  - FeinFocus 160 kVp
  - 10 micron focal spot

- **The detector:**
  - Thompson TH9438HX image intensifier
  - Retiga 1024x1280 10-bit CCD, lens coupled

- **The sample stage:**
  - Newport RV120 high load rotational stages, opposed
  - Instron 4444 load frame, 2000N capacity
Strain Measurement - Details

Strain Tensors Calculated at Each Point From Groups of Displacement Vectors

Displacement Vectors Measured at Many Points Throughout a Sample

2005 SWST/FPS, Quebec City, Canada
Software Details

- **Image collection:**
  - Field of view between .5 and 10 cm
  - 1000 projections (360 degrees)

- **Reconstruction:**
  - Feldkamp-style filtered back-projection
  - ~ 1 billion voxels, res. range 5 - 100 µm

- **Data volumes:**
  - Projection images ~ 2GB, reconstruction volumes ~ 1 GB
  - Collection ~ 20 min (PC/GPIB based)
  - Reconstruction (2 vols) ~ 14 hrs (2 proc Sun)
MATERIAL
Component Materials

- Wood flour
  - 40 mesh pine from American Wood Fibers
  - Oven dried prior to use for 24 hours at 103°C

- Plastic
  - High Density Polyethylene: BP Solvay B53 35H FLK, melt flow = 0.49 g/10 min

- X-ray attenuation contrast enhanced by doping the matrix with chemically inert gold nano-spheres
PARTICLE CHARACTERIZATION

Particle Area Distribution

Particle Aspect Ratio Distribution

2005 SWST/FPS, Quebec City, Canada
## Phase Characteristics

### Density profiles

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wood cell</td>
<td>1.5</td>
</tr>
<tr>
<td>HDPE</td>
<td>0.95</td>
</tr>
<tr>
<td>PVC</td>
<td>1.3 to 1.58</td>
</tr>
<tr>
<td>PP</td>
<td>0.9</td>
</tr>
</tbody>
</table>

Wood particles and the polymer matrix material have similar densities and x-ray absorption levels (CHO).
**GOLD NANOSPHERES**

HAuCl$_4$ in water

(C$_8$H$_{17}$)$_4$N$^+$ Br$^-$

Two phase system: water/toluene

Gold moves to toluene phase
Remove aqueous phase

Metallic gold precipitates as spheres, 2 – 5 nm diameter

NaBH$_4$

Centrifuge to remove gold nano-spheres
Add to HDPE in Brabender Plasticorder mixer

2005 SWST/FPS, Quebec City, Canada
Enhanced CT contrast

2005 SWST/FPS, Quebec City, Canada
Enhanced CT contrast

1% Au  0.1% Au  Ref

1% Au

Reconstruction of a longitudinal section
20 µm/voxel

2005 SWST/FPS, Quebec City, Canada
Enhanced CT contrast: with a price tag...

- **MOE [GPa]**
  - Control
  - 0.1% gold
  - 1% gold

- **MOR [MPa]**
  - Control
  - 0.1% gold
  - 1% gold

- **Density [g/ccm]**
  - Control
  - 0.1% gold
  - 1% gold

- **Property change comparison**
  - MOE
  - MOR
  - Density
  - Control
  - 0.1% gold
  - 1% gold
Blending

Brabender
Intelli-Torque
Plasticorder

☐ Melt HDPE at 170° C
☐ Add gold n-spheres
☐ Add wood flour
☐ Mix 10 min
☐ Remove and store for compression molding

2005 SWST/FPS, Quebec City, Canada
Pressure molding

Carver Press

- Mold: 101.6 x 101.6 x 2 mm
- Temperature: 185°C
- Preheat time: 10 min
- Press time: 10 min
- Press pressure: 344.8 kPa
- Cooling pressure: 344.8 kPa
Specimens

- 10 specimens for each wood-PE composite board

- Specimen size
  - L: 54.5 $\pm$ 2.0 mm
  - W: 12.5 $\pm$ 2.0 mm
  - (w: 9.2 $\pm$ 0.1 mm)
  - t: 2.6 $\pm$ 0.5 mm
Static tensile tests

2005 SWST/FPS, Quebec City, Canada
Static tensile tests

<table>
<thead>
<tr>
<th></th>
<th>E* (MPa)</th>
<th>UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>1755</td>
<td>14.80</td>
</tr>
<tr>
<td>2</td>
<td>1934</td>
<td>14.68</td>
</tr>
<tr>
<td>3</td>
<td>1527</td>
<td>12.71</td>
</tr>
</tbody>
</table>

*linear fit to a section of non-linear data
Static tensile tests: nonlinearity

<table>
<thead>
<tr>
<th>#</th>
<th>$E_{\text{tan}*}$ (MPa)</th>
<th>$E_{1%*}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3131</td>
<td>487</td>
</tr>
<tr>
<td>2</td>
<td>3846</td>
<td>481</td>
</tr>
<tr>
<td>3</td>
<td>2776</td>
<td>387</td>
</tr>
</tbody>
</table>

*from exponential fit to $E(\varepsilon_{yy})$ data*
Static tensile tests: nonlinearity

<table>
<thead>
<tr>
<th></th>
<th>$E_{\text{tan}}$ *</th>
<th>$E_{\text{sec1%}}$</th>
<th>$E_{\text{sec2%}}$</th>
<th>$E_{1%}$ *</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>MPa</td>
<td>MPa</td>
<td>MPa</td>
<td>MPa</td>
</tr>
<tr>
<td>1</td>
<td>3131</td>
<td>1221</td>
<td>721</td>
<td>487</td>
</tr>
<tr>
<td>2</td>
<td>3846</td>
<td>1242</td>
<td>731</td>
<td>481</td>
</tr>
<tr>
<td>3</td>
<td>2776</td>
<td>1082</td>
<td>629</td>
<td>387</td>
</tr>
</tbody>
</table>

*from exponential fit to $E(\varepsilon_{yy})$ data

2005 SWST/FPS, Quebec City, Canada
Static tensile tests: Poisson effect

*linear fit to a section of experimental data (0.5% - 2.5%)

<table>
<thead>
<tr>
<th>#</th>
<th>$\nu_{0%}$</th>
<th>$\nu_{2%}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-0.3516</td>
<td>-0.2959</td>
</tr>
<tr>
<td>2</td>
<td>-0.3597</td>
<td>-0.3033</td>
</tr>
<tr>
<td>3</td>
<td>-0.3412</td>
<td>-0.2805</td>
</tr>
</tbody>
</table>

2005 SWST/FPS, Quebec City, Canada
Degrading procedure

Degradation Procedure, Spec#09

Load, N

time, s

0 120 240 360 480 600 720 840 960

0 100 200 300 400

2005 SWST/FPS, Quebec City, Canada
Degrading procedure

![Graph of stress vs. avg strains for WPC #1 & #6](image)

2005 SWST/FPS, Quebec City, Canada
max = 0.015
max = 0.020
max = 0.035

Consistent weak areas...

350 N
375 N
ultimate
Degrading procedure
Work in progress...
Acknowledgement

This research is funded by the OSU General Research Fund Award, Fall 2004.
Questions?
Preliminary Conclusions

- Digital images carry wealth of quantitative information...

- Find an alternative contrast enhancing treatment