Evaluation of Durability in Wood-Plastic Composites Using X-ray Tomography

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Outline

- Background and introduction to XMT
- Experimental setup
- Results
- Conclusions
- Acknowledgements
Wood flour in HDPE
Wood-Plastic Composites (WPCs)

- Mechanical and physical properties of WPCs 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 formulation, processing and coupling agents
  - Improve mechanical properties
  - Improve water sorption and durability
How much can flexural tests reveal about internal bonding?
WPC micromechanics

- Durability and mechanical performance of WPCs are decided on the µ-mechanical level - in the interphase between wood and plastic

- Traditional testing methods offer indirect and limited insight to µ-mechanical performance, µ-damage accumulation and governing failure mechanisms – new methods are needed

- 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

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OBJECTIVE

To develop experimental procedures for multi-scale evaluation of micro-mechanical performance, governing failure mechanisms and micro-damage accumulation in WPCs
Experimental questions

- Where does the damage take place?
  - Statistical characterization of the local deformation and internal damage accumulation
- How bad is it?
  - Correlations between the internal damage and the storage modulus
- When will it happen?
  - Accelerated aging
  - Samples from the field
  - Predictive models
Procedure: Multi-scale analysis

- Macro level: elastic modulus, static strength, storage modulus, original and aged (meters)

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

- Digital Volume Correlation based on low resolution CT scans (20 µm/pxl) to identify internal strain concentration areas of interest

- High Resolution CT scans (3 µm/pxl) on those areas 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?

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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 (2D)
- Out-of-plane deformations from multiple-camera surface images (3D, Poisson’s ratio)
- 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
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Integrated Testing and Imaging

Detector | Sample Stage | X-ray Source
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
Software Details

- **Image collection:**
  - Field of view between .5 and 10 cm
  - 1000 projections (360 degrees) @ 4 sec/ea

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

- **Data volumes:**
  - Projection images ~ 2GB, reconstruction volumes ~1 GB
  - Collection ~ 3 h (PC/GPIB based)
  - Reconstruction (2 vols) ~14 hrs (2 Sun processors)
Volumetric Strain Measurement – How it works

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
Strain Measurement - Details

Strain Tensors Calculated at Each Point From Groups of Displacement Vectors

Displacement Vectors Measured at Many Points Throughout a Sample

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INITIAL EXPERIMENTS
Component Materials

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

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

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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
PARTICLE CHARACTERIZATION

Particle Area Distribution

Particle Aspect Ratio Distribution

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Specimens

- 10 specimens for each wood-PE composite board
- Specimen size
  - L: 54.5 ±2.0 mm
  - W: 12.5 ±2.0 mm
  - (w: 9.2 ±0.1 mm)
  - t: 2.6 ±0.5 mm
Wood particles and the polymer matrix material have similar densities and x-ray absorption levels (CHO).
GOLD NANOSPHERES

HAuCl₄ in water

(C₈H₁₇)₄N⁺ Br⁻

Two phase system: water/toluene

Gold moves to toluene phase
Remove aqueous phase

NaBH₄

Metallic gold precipitates as spheres, 2 – 5 nm diameter

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

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Enhanced CT contrast: with a price tag...
Enhanced CT contrast
Enhanced CT contrast

Reconstruction of a longitudinal section
20 µm/voxel

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Static tensile tests
Static tensile tests

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

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

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Static tensile tests: nonlinearity

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*from exponential fit to $E(eyy)$ data
Static tensile tests: Poisson effect

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

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Degrading procedure

Degradation Procedure, Spec#09

load, N

0 100 200 300 400

0 120 240 360 480 600 720 840 960
time, s

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Degrading procedure

![Graph showing stress vs. average strain for WPC #1 & #6.](image)
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Degradation Procedure, Spec #09

Load, N

Time, s

Consistent weak areas...

strain

max = 0.015

max = 0.020

max = 0.035

350 N

375 N

ultimate

window 15x15
Degrading procedure
Digital Volume Correlation of WPCs

☐ Just getting started
XRT 3D volumetric reconstruction of gold-modified 40% wood/HDPE composite
Gold and holes
Gold and holes
Work in progress...
Acknowledgement

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