

# JMFA3—Confirmation Study Looking into Rotation and Tilt Effects on X-Ray Diffraction Microfibril Angle Estimation

X-ray diffraction techniques have the potential to dramatically decrease the time required to determine microfibril angles (MFAs). The latest version of a Javabased curve-fitting tool, JMFA3, permits us to reduce the time required to evaluate MFA x-ray diffraction patterns. Because this tool reflects the underlying physics more accurately than do existing tools, we expect it to yield more accurate estimates of MFA.

## **Background**

Much of our future timber supply is expected to come from improved softwood and hardwood trees grown on managed plantations or from small-diameter timber removed during forest management operations. This short-age-rotation resource will contain higher proportions of juvenile wood than the present resource.

Juvenile wood has substantially lower mechanical property values than mature wood, which generally accounts for the inferior properties of short-rotation plantation wood. These reductions in properties may be mitigated through improved silvicultural practices, which require improved tools for monitoring stand quality. Measuring MFA holds promise as one such tool.

Wood cells are made up of multiple layers: a primary layer (P) and three secondary layers (S1, S2, S3). The thickest of the secondary layers is the S2 layer, and its properties strongly influence the properties of the wood fiber. MFA is the angular deviation from the vertical of the microfibrils in the S2 layer (Fig. 1).

Orientations of the microfibrils in the S2 layer of juvenile wood tracheids vary widely both within and among trees of a species. There is strong evidence that the MFA of the S2 layer of the woody cell wall is a critical factor in the mechanical behavior of wood, and MFA is

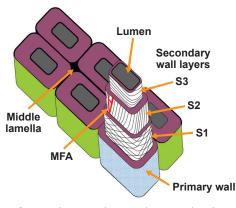


Figure 1.
A schematic of wood cell structure with the MFA identified.

a factor that can be used as a selection tool in plantation management.

Verrill et al. (2001) developed a curve fitting tool to estimate MFA from x-ray diffraction patterns obtained from small wood specimens. Simulation experiments suggest that the tool does an excellent job of estimating MFA, especially for larger MFA (Verrill et al. 2006). On a large sample of specimens, they also obtained relatively good agreement between their x-ray-based microfibril angle measurements and measurements obtained via a much slower and likely less accurate visual light microscopy method. They recently developed a new version of their computer program and want to subject it to additional tests. The second and third generations of their theory/computer program estimate specimen tilt and rotation as well as microfibril angle from x-ray diffraction patterns. To further validate the theory/computer program, x-ray diffraction measurements must be performed on wood specimens (for which microscopic estimates of microfibril angle are also available) subjected to a series of controlled rotations and tilts

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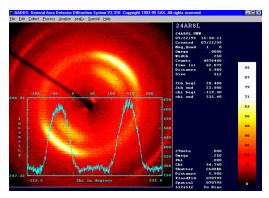


Figure 2.
An example of the x-ray diffraction intensity pattern produced by the Siemens Hi-Star x-ray diffraction device.

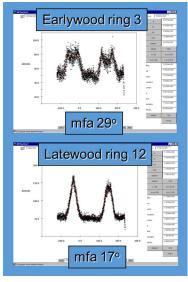
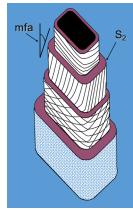


Figure 3. Images from the JMFA curve fitting software.



## **Objective**

The objective of this project is to obtain additional empirical evidence for the theoretical relationship between a specimen's microfibril angle, rotation, and tilt and the corresponding x-ray diffraction pattern. If the mathematics underlying the program accurately reflects the physics behind the x-ray diffractions, the program should be able to correctly estimate specimen rotations and tilts (known in the experiment). Also, MFA estimates for a particular specimen should be stable even in the presence of a variety of controlled rotations and tilts.

# **Approach**

Testing will be conducted on the Siemens Hi-Star x-ray diffraction area detector system using the Siemens General Area Detector Diffraction Software (GADDS) to determine intensity profiles corresponding to MFA orientation of cell walls (Figs. 2 and 3) of fifteen specimens with "known" (via microscopy) MFAs. Five specimens will have small MFAs (approximately 10°),

five will have intermediate MFAs ( $25^{\circ}$  to  $30^{\circ}$ ), and five will have large MFAs ( $40^{\circ}$  to  $50^{\circ}$ ). X-ray diffraction patterns will be obtained at  $5^{\circ}$  rotation increments from a nominal  $0^{\circ}$  to a nominal  $180^{\circ}$ . In addition, patterns will be obtained at  $10^{\circ}$ ,  $20^{\circ}$ , and  $30^{\circ}$  tilts.

#### **Expected Outcomes**

This research program should provide test results that support the use of the new software developed by FPL scientists as a fast and accurate estimator of MFA.

#### **Timeline**

The project was initiated in June 2016. Testing should be completed by November 2016, and the final report and recommendations will be completed by June 2017.

#### Cooperators

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#### References

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