Measuring The Tensile Strain of Wood By Visible And Near-Infrared Spatially Resolved Spectroscopy

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Te Ma: methodology, data collection and analysis, writing-original draft preparation

Tetsuya Inagaki: methodology, data validation, and writing-reviewing.

Masato Yoshida: contributed to capture the submicroscopic changes during tension testing, writing-reviewing

Mayumi Ichino: data collection and analysis

Satoru Tsuchikawa*: supervision, conceptualization, writing-reviewing and editing

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Abstract:

Wood has various mechanical properties, so stiffness evaluation is critical for quality management. Using conventional strain gauges constantly is high cost, also challenging to measure precious wood materials due to the use of strong adhesive. This study demonstrates the correlation between light scattering changes inside the wood cell walls and tensile strain. A multifiber-based visible-near-infrared (Vis–NIR) spatially resolved spectroscopy (SRS) system was designed to rapidly and conveniently acquire such light
scattering changes. For the preliminary experiment, samples with different thicknesses were measured to evaluate the influence of thickness. The differences in Vis–NIR SRS spectral data diminish with an increase in sample thickness, which suggests that the SRS method can successfully measure the whole strain (i.e., surface and inside) of wood samples. Then, for the primary experiment, 18 wood samples with the same thickness (2 mm) were tested to construct a strain calibration model. The prediction accuracy was characterized by a determination coefficient ($R^2$) of 0.86 with a root mean squared error (RMSE) of 297.89 με for five-fold cross-validation; for test validation, the prediction accuracy was characterized by an $R^2$ of 0.82 and an RMSE of 345.44 με.

Keywords:
Tensile strain of wood, non-destructive evaluation, spatially resolved spectroscopy, visible and short-wave light scattering, multivariate analysis

1. Introduction

Wood is a natural material with multi-layered elongated cells. Due to the variability of its mechanical properties, the stiffness evaluation of each structural wooden member is critical for quality management (Baillères et al. 2012; Montero et al. 2012). The wood
cell wall is a macromolecular composite formed of cellulose, hemicelluloses, and lignin (Hon and Chang 1984). Cellulose is the primary component in bearing tensile stress (Salmén and Bergström 2009). In contrast, hemicelluloses function as a coupling agent to hold the cellulose (Burgert 2006). The conventional method for wood strain measurement is to use a strain gauge, which is high cost (either disposable or reusable ones) in constant use (Yang et al. 2005). In addition, it is challenging to measure precious wood materials due to the use of a strong adhesive, which can destroy the wood after removal. For example, the heritage community generally does not allow the application of strain gauges on wooden arts (Anaf et al. 2020). Moreover, difficulties arise when strain gauges are used in an environment where the electromagnetic wave interference is extensive (Liu et al. 2015; Barr et al. 2017).

X-ray diffraction (Kamiyama et al. 2005) and infrared (IR) spectroscopy (Åkerholm and Salmén 2001) can detect wood structure-function relationships at the nano- and microstructural levels. Indeed, Raman spectroscopy (Hsieh et al. 2008) can predict sample strains based on a typical shift peak at a wavenumber of 1095 cm$^{-1}$, which corresponds to the glycosidic bond in the cellulose structure backbone (Guo and Altaner 2018). Moreover, the band shift varies among specimens (Eichhorn 2001), and the
molecular strain is smaller than the macroscopic strain (Peura et al. 2007). In contrast, Near-IR (NIR) spectroscopy (wavelength: 800–2500 nm or wavenumber: 12500–4000 cm\(^{-1}\)) is well-suited for characterizing organic compounds in samples, mainly in combination with multivariate mathematical techniques. When NIR light illuminates and transmits through an object, the energy of the incident electromagnetic wave changes due to the stretching and bending vibrations of chemical bonds, such as O–H, N–H, and C–H. Subsequently, the quality and quantity of an object can be evaluated nondestructively, rapidly, and cost-effectively by analyzing the light reflectance and transmittance values (Ma et al. 2020). Compared with microtomed sections needed for IR spectroscopy, NIR spectroscopy can nondestructively measure wood samples up to several millimeter thick without special sample pretreatments (Salmén and Bergström 2009). This is essential when focusing on practical applications, as thin samples prepared in lab behave differently than solid wood, e.g., including stress relaxation in the former (Yu et al. 2009). Taking advantages of NIR spectroscopy, Guo et al. (2019) analyzed band shifts and band assignments on NIR light absorbance characteristics during the wood tension test, the results of which suggest that the observed band shifts correlate with wood tension levels. Their study is impressive and has reference value for future use of NIR spectroscopy. However, several issues can be highlighted, such as small-signal band shifts on the
collected NIR spectra; e.g., a sample strain of 0.1% would induce a band shift of approximately 2 cm$^{-1}$ at around 6470 cm$^{-1}$ ($\text{3OH} \cdots \text{5O}$). Moreover, since the molecules are influenced by neighboring molecules, actuarial peaks overlap on the NIR spectra (Okazaki 2012). Indeed, advanced curve fitting approaches could be used to predict the peak shift, but the band shift varies among specimens, which has been confirmed by the IR method (Eichhorn 2001). The spectral information of such long-wave sensitive spectrometers is rather rich, and, as such, it requires expensive equipment, such as detectors and light sources (Xing et al. 2008). Accordingly, there is still room to develop and improve NIR spectra collection and data analysis methods, especially for practical application purposes.

The most likely to be neglected is that bulk wood is a highly scattering medium, and the light scattering degree inside the wood cell wall also highly correlates with the microstructure (Ban et al. 2018; Ma et al. 2018a, 2019). Studies show that the reduced light scattering coefficient ($\mu'_s = 10 - 100 \, \text{cm}^{-1}$) is much larger than the absorption coefficient ($\mu_a = 0.05 - 1.00 \, \text{cm}^{-1}$) in the wavelength range of 700-1040 nm for both softwood and hardwood species treated in different ways (dry, wet and degraded) (D’Andrea et al. 2007). The deformation under longitudinal tension includes
macromolecule deformations in the layers and interlaminar slippages. The former is related to the structure, orientation, and interaction of the polymers in the wood, and the latter slippage deformation results from the structural differences between cell-wall layers (Keckes et al. 2003; Adler and Buehler 2013). The misalignment between the cellulose fibrils to the strain direction is amplified by bending and shearing of anatomical structures and straightening of slightly waved cellulose microfibrils, forming lenticular spaces between adjacent fibrils (Salmén 2015). The weak interfaces of wood cells or annual rings could deflect transverse cracks into the longitudinal plane (Smith et al. 2003; Marthin and Kristofer Gamstedt 2019; Guo et al. 2020). Hence, effective utilization of the light scattering degrees (i.e., microstructure changes) inside the wood during tension testing should predict strain levels accurately. This method also can reduce costs associated with equipment because shorter wavelengths are scattered more strongly than longer wavelengths in the visible (Vis)–NIR optical range (Ma et al. 2018a).

However, conventional Vis–NIR spectrometry acquires spectral data from a single sample point based on the collective effects of light absorption (due to chemical components such as water and cellulose content) and scattering (due to physical structures such as cell size and intercellular spacing) (Vanoli et al. 2020). Studies have mainly relied
on performing further spectral pretreatments, such as baseline offset correction or standard normal variate (SNV), that reduce light scattering effects before training calibration models (Zude et al. 2011). Spatially resolved spectroscopy (SRS) requires relatively strong, steady-state spotlights for illumination, and its diffusely reflected light pattern is collected at multiple distances for light absorption and scattering evaluation (Farrell et al. 1992; Qin et al. 2009; Lu et al. 2020). SRS has two main measurement configurations: spectral imaging and fiber probing. On the one hand, the SRS based on spectral imaging is a non-contact method that measures spatially resolved diffuse reflectance over a broad spectral range (Peng and Lu 2008; Qin and Lu 2008; Zhu et al. 2015). The measurement system mainly consists of a hyperspectral imaging (HSI) camera, a prime lens, and a small broadband beam as illumination. However, the light beam and source-detector distance need to be carefully considered in this configuration, as they determine the measured results (Cen and Lu 2010; Lu et al. 2020). On the other hand, the fiber probe-based SRS is a contact method, which is often inconvenient for rapid online quality assessment (Ma et al. 2018a). However, due to the easy-to-operate design with a strong light reflectance, fiber probe-based SRS portable systems are desirable alternatives for on-site applications. Besides, contact methods are more suitable to predict the strains of moving wood samples caused by tension testing than non-contact methods.
This paper reports on wood strain prediction results obtained by evaluating the changes in Vis–NIR SRS spectral data collected from wood samples during tension testing. The objectives of this paper are as follows: (1) design a multifiber-based Vis–NIR SRS system to rapidly and nondestructively acquire light scattering characteristics; (2) examine the relationship between SRS signals and wood tension strains by principal component analysis (PCA); and (3) construct wood strain calibration models by partial least squares (PLS) regression. This study should provide new insights into SRS methods for practical application to predict the tensile strain of wood conveniently and cost-effectively.

2. Materials and methods

2.1 Sample preparation

Wood samples (*Hinoki cypress*) with a length of 120 mm (longitudinal), a width of 10 mm (radial), and various thicknesses (tangential: 2 mm, 3 mm, 4 mm, and 5 mm) were sawn from air-dried wood board commercially purchased from a local wood processing company. Specimens were selected from mature wood parts sufficiently far from the pith to neglect ring curvature.

For the preliminary experiment, samples with different thicknesses (2 mm, 3 mm, 4 mm, and 5 mm) were measured to evaluate the influence of thickness. Then, for the
primary experiment, 18 wood samples with the same thickness (2 mm) were tested to construct a strain calibration model. The samples were selected based on the wood fiber directions, which were as parallel as possible to the longitudinal direction. Before the experiment, all the samples remained in a desiccator, where relative humidity (RH) was controlled at 59% with a saturated salt solution of sodium bromide. Subsequently, the sample weights were measured for the equilibrated specimens using a digital balance (accuracy of 0.0001 g). A digital caliper (0.01 mm accuracy) measured the sample dimensions under the equilibrated condition. From the measured weights and dimensions of the raw data, sample equilibrium moisture content (EMC) and density were calculated according to the following equations:

\[
\text{EMC}(\%) = \frac{W - W_d}{W_d} \quad (1)
\]

\[
\text{Density} \left( \frac{\text{kg}}{\text{m}^3} \right) = \left( \frac{W}{V} \right) \quad (2)
\]

where \( W \) and \( W_d \) are weights for the equilibrated and oven-dried samples, respectively, and \( V \) is the sample volume under the equilibrated condition.

**2.2 Tensile test**

Each prepared wood sample was placed in a bending testing machine (preliminary experiment: Shimadzu AG-100KNI, Shimadzu, Japan; primary experiment: The SVZ-
During tension test, the bending machine was suspended several times manually to obtain strain measurements and Vis–NIR SRS data. The strain was recorded with strain gauges (FLAB-5-11, Tokyo Sokki Kenkyujo, Japan) glued to one side of each sample with instant adhesive (CN, Tokyo Sokki Kenkyujo, Japan) and connected to a strain-meter (TC-32K, Tokyo Sokki Kenkyujo, Japan). A Vis–NIR measurement system was used to collect light scattering characteristics on the other sample side (Fig. 1 (a) and (b)).

2.3 Visible and near-infrared spatially resolved spectroscopy measurements

Fig. 1 (c) and (d) show the measurement part (i.e. the fixator of light illumination and detection fibers) of the proposed Vis–NIR SRS system and a diagram of the internal structure. A 5-W halogen lamp initially provided light illumination. An optical fiber (SOG-70S, Sumita Optical Glass, Inc., Saitama, Japan) translated the light source onto each wood sample. Then, 30 silica fibers (Vis–NIR type, Core: 100 μm, Clad: 110 μm, Fiberguide Industries, New Jersey, USA) were separated into five groups (1, 2, 3, 4, and 5 from the light illumination point) to collect the diffuse reflected light and transfer it to the Vis–NIR HSI camera (SPECT-100nir1F, Spectral Application Research Laboratory Co., Ltd. Shizuoka, JAPAN). Inside the camera, the light beam was dispersed into spectral
components (vertical axis) while preserving spatial information (horizontal axis), and the camera took two-dimensional light signals. The shutter speed and framerate were set at 15 ms and 8 fps, respectively. In this study, the fixator was pasted parallelly to the sample grain direction with a double-sided tape, which was easily removable after measurement collection. The sensitive wavelength range of the Vis–NIR HSI camera was 600–1100 nm, with a minimum reading width of approximately 0.65 nm/pixel and a spectral resolution of 4.5 nm. For each measurement, 16 spectral images were captured and their averaged values saved. Light reference was measured using a tailor-made integrating sphere, i.e., a plastic ball (diameter of 6 cm) covered in barium sulfate. Dark values were measured by turning off the light and covering the light-collection fibers. Wavelengths under 660 nm and over 1002 nm were noisy and unreliable. Thus, a wavelength range of 660–1002 nm was selected for further data analysis. The collected spectra were then converted to the reflectance values by Eq. (3):

\[
R_\lambda = \frac{S_\lambda - D_\lambda}{B_\lambda - D_\lambda}
\]

where \( \lambda \) denotes the wavelength, \( S \) and \( B \) are the sample and a white reference spectrum, respectively, and \( D \) is the dark spectrum.
A digital camera took photos (16 × amplification) of another wood sample (thickness of 2 mm) before and after the tensile test to understand the submicroscopic changes during tension testing.

**Fig. 1** (a) Experiment instruments; (b) Vis–NIR SRS data and strain measurements; (c) fixator of the Vis–NIR SRS measurement system; (d) internal structure diagram of the fixator.

### 2.4 Spectra pretreatments and principal component analysis

The Vis–NIR SRS spectra were smoothed by a Savitzky–Golay filter (polynomial order: 2; frame length: 15). The spectral data, collected before tension testing subtracted from various strain levels, were tested to correct the natural variability of physical structure among wood samples. Then, PCA with the mean center was used to “compress” the Vis–NIR spectral data to examine the correlation between measured strain reference values and the spectra data changes. It is noteworthy that no other spectra pretreatments
(e.g., SNV (Cuesta Sánchez et al. 1995) and the second derivative (Gorry 1991)) were used in this study to keep the maximum light scattering information.

2.5 Partial least squares regression analysis

The Vis–NIR difference spectral data were calibrated with the measured strain values via PLS regression (Martens and Tormod 1992). In developing the PLS regression models, 70% of measured data was randomly selected as the calibration set, leaving 30% for the test set. Five-fold cross-validation optimized the number of latent variables (LVs). The coefficients of determination ($R^2$) and the root mean squared error (RMSE) characterized the constructed calibration model’s performance:

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(y_i - \hat{y}_i)^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2}$$

$$\text{RMSE} = \sqrt{\frac{1}{n} \sum_{i=1}^{n}(\hat{y}_i - y_i)^2}$$

where $n$ is the number of measurements, $y$ is the reference strain values, $\hat{y}$ is the strain value predicted by PLS regression analysis, and $\bar{y}$ is the mean value of $y$.

Variable importance in projection (VIP) scores were used to identify the influence of the SRS spectral data collected by the individual fiber groups on the PLS model (Wold et al. 1993; Farrés et al. 2015). Data analysis was performed by MATLAB (The MathWorks Inc., Natick, MA).
3 Results and discussion

Fig. 2 shows the raw spectral image of a wood sample with a thickness of 2 mm taken by the Vis–NIR HSI camera before the tensile test. This image data contains the spatial information of the 30 silica fibers (horizontal axis) and the spectral information of the measured wood sample (vertical axis). The main difficulty with conventional SRS methods is associated with collecting the spectral data with a high signal-to-noise ratio quickly. One method involves repeating the same data measurements and averaging the results, which is time-consuming (Tkachenko 2006). This study is desirable for the spectral data acquisition time to be short of excluding additional relaxation phenomena (Burgert 2006; Altaner et al. 2014) This was achieved by a two-step signal averaging process: (i) each fiber occupies 34 pixels of the HSI camera, and the central 30 pixels were averaged for spectral data collection, after which (ii) the signals of six fibers in the same group were averaged.
Fig. 2. Raw Vis–NIR SRS spectral image of wood sample with 2 mm thickness.

Fig. 3 shows the Vis–NIR SRS spectra with standard deviations of the 18 wood samples at various tension levels, which were initially calculated from the raw spectral image shown in Fig. 2. It is logical that the overall spectral intensity quickly falls with an increase in distance from the light illumination. The wavelength at 925 nm corresponds to the third overtone of C–H absorption (Mohammadi-Moghaddam et al. 2018), which can be attributed to the chemical components of the wood samples. The wavelength at approximately 930 nm has the highest light reflectance when the fiber group is 3–4 mm away from the light illumination, suggesting that the light at said wavelength was less absorbed and transmitted further from the light illumination than other wavelengths along the wood grain direction. It is noteworthy that the optical scattering was not isotropic within each wood sample; the light propagated further in the parallel direction.
because the scattering coefficient along the cylinders is much smaller than that in the perpendicular direction to the cylinders (Ma et al. 2018b, 2019).

Fig. 3 Averaged Vis–NIR raw spectra and the standard deviations (light color) of 18 samples with the same thickness (2 mm) and various tension strains.

Fig. 4 shows the Vis–NIR spectral data for various strain measurements for wood samples with different thicknesses (vertical) by the five fiber groups (horizontal), respectively. The wavelength range was selected to 900–950 nm to expand the image size. The light reflectance increased with an increase in wood strain. Light absorption at 925 nm is the most obvious at the spectra collected by the 1-mm fiber group. The signal quality decreases with an increase in distance between the light illumination and light-detection fibers, suggesting that different fiber groups can collect spectral data with
different light absorption and scattering degrees. Light reflectance is also affected by sample thickness. The differences in the Vis–NIR spectral data, caused by sample strains, diminished in thicker samples, especially at far fiber groups. This could be due to the light transmission depth is different among the wood samples at various thicknesses. In this study, the Vis–NIR light was transmitted through hinoki wood samples with an approximate thickness of 5 mm (see the supplementary, Fig. S1), after which it was reflected by the white plate and detected by optical fibers. However, because thicker wood samples have a more profound light transmission, which affected light propagating in parallel, stronger noise was associated with the collected spectra at more extended fiber groups. Except for signal quality, because the strain gauge was stuck on the opposite side of the SRS fixator, less transmission light could also reduce the correlation between SRS data and strain reference values. This suggests that the proposed SRS equipment is suitable for predicting the tension strain of wood samples with an approximately of 2–3 mm. Further improvements could be useful to construct strain prediction models for thicker samples, such as reducing the distance between fiber groups and introducing a method to measure the strain changes where the spectral data were collected.
Fig. 4 Vis–NIR SRS spectral data collected at various sample thicknesses (vertical) by different fiber groups (horizontal).

Fig. 5 (b) and (c) show the digital photos of wood samples before and after tension testing with a strain of 3410 με. The stretched wood cell wall could decrease the density of the measured area and increases the aperture on the cell wall between the light source and the five fiber groups of the SRS fixator. Hence, the light reflectance values increased during the tension test.

Fig. 5 Digital photos of the wood sample with 2 mm thickness before (a) and after (b) the tension test, respectively.

The spectral data collected from the five fiber groups at a wavelength range of 660–1002 nm were concatenated, resulting in 2555 variance values (Figure 6 (a1–i)). Fig. 6 (b1–i)) shows the first two principal component (PC) loadings. The PC loadings can be understood as the weights for each variance value when calculating the PC score. The
accumulated contribution rate of the first two scores is approximately 99.64%, 99.15%, 97.47%, and 94.48% for a sample thickness of 2, 3, 4, and 5 mm, respectively. The PC1 and PC2 scores of the SRS are shown in Fig. 6 (c1-4), where the Y-axis shows PC2 and the X-axis shows the PC1. There is a strong correlation between PC1 loading and light scattering differences, i.e., vertical baseline shift. As expected from Fig. 4, the contribution rate of the PC1 score decreases with an increase in sample thickness. Moreover, PC2 loading has relatively high absolute values at a light wavelength of 970 nm. This suggests a meaningful correlation between the light absorption by hydrogen bonds and wood strain changings, but the contribution rate was much lower than the light scattering differences.
Fig. 6 (a1-4) Concatenated Vis-NIR difference spectral data used for PCA; (b1-4) first two principal component loadings; (c1-4) first two principal component scores of the concatenated Vis-NIR spectral data.

Fig. 7 (a) shows the strain calibration results of the wood samples with the same thickness (2 mm) from the PLS regression method with LV numbers 9 (explanatory...
variables: Vis-NIR difference spectral data after pretreatments; response variables: strain reference values). The density of the samples was $415 \pm 14$ kg/m$^3$ and the MC was $10.51\% \pm 0.26\%$. Overall, the PLS calibration model has a high prediction accuracy: the $R^2$ and RMSE of the calibration set are 0.86 and 297.89 $\mu e$, respectively. For the validation set, the $R^2$ and RMSE are 0.82 and 345.44 $\mu e$, respectively. Differences in the RMSE can be attributed to the fact that the strain gauge only measured the surface strain of each wood sample, whereas the SRS method measured the whole strain with the information of sample thickness. Indeed, there is a possibility that the SRS method can more accurately measure the wood sample strain than conventional strain gauges, but further studies are required to prove this. Overall, this is the first study to suggest that the Vis–NIR SRS method combined with PLS regression analysis can predict the tensile of wood samples with the same thickness (also see the prediction results of the wood samples with the same 5 mm thickness in supplementary, Fig. S2). Fig. 7 (b) shows the strain calibration results when using the raw Vis–NIR spectral data, it is evident that the difference spectral data could be used to achieve the initial value correction purpose, thus are necessary to construct the strain calibration model.
Fig. 7 Scatter plot of measured and predicted strain values using (a) difference spectral data or (b) raw spectral data.

Fig. 8 (a) shows the VIP scores, from which it is evident that the number of important wavelengths (VIP scores>1) decreased with an increase in distance between light detection and illumination. Fig. 8(b) shows the PLS regression coefficients regarding the Vis–NIR difference spectra. It was somewhat surprising that although the contribution rate calculated by PCA at the wavelengths related to the hydrogen bonds is much lower than the light scattering differences at each sample, it is important to construct a strain calibration model using all 18 samples. It could be supported by the knowledge that wood becomes more ductile with increased MC (Ozyhar et al. 2012; Mvondo et al. 2017) which also affects the light scattering degree (Konagaya et al. 2016). This also suggests that MC effects much be fully valued, to build individual calibration models depends on sample MC may be the best way to reduce the MC effects.
Conclusion

This study aims to demonstrate the correlation between light scattering changes inside the wood cell walls and tensile strain. Spatially resolved diffuse reflectance was collected by designing a portable and cost-effective measurement system based on fiber probes. For the preliminary experiment, samples with different thicknesses (2 mm, 3 mm, 4 mm, and 5 mm) were measured to evaluate the influence of thickness. Then, for the primary experiment, 18 wood samples with the same thickness (2 mm) were tested to construct a strain calibration model. The prediction accuracies were characterized by an $R^2$ of 0.86 and an RMSE for 297.89 $\mu$ε for five-fold cross-validation, 0.82 and 345.44 $\mu$ for test validation. The designed SRS measurement system does not require sophisticated measurement techniques. Moreover, it has a cost design effective design due to the Vis–
NIR HSI camera with short-wave sensitivity, which is much cheaper than long-wave sensitivity cameras.

Further research should focus on extending the applicability of the SRS approach to a broader database of wood types and larger sample numbers with various thicknesses. The intervals between fiber groups could be changed to test the strain prediction of thicker wood samples. This research also references further research to measure growth strain in trees non-destructively. However, because light scattering degree is also affected by MC, this would require more in-depth spectral pretreatments.

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