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繊維方向一軸応力を受ける木材のマクロなひずみと結晶格子ひずみとの関係

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The Relationship between Macroscopic Strain and Crystal Lattice Strain in Wood under Uniaxial Stress in the Fiber Direction

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Abstract

This study investigated the relationship between the cellulose crystal lattice strain (crystalline region) and the macroscopic surface strain in specimens of *Chamaecyparis obtusa* wood under uniaxial stress in the fiber direction. Changes in the strain of the crystal lattice were measured from the peak of (004) reflection using the transit X-ray method. The macroscopic surface strain of each specimen was measured with a strain gauge.

1) Up to a stress level of about 35%, the dynamic behavior of the crystalline region in the wood showed very similar qualitative tendencies under uniaxial compressive stress and uniaxial tensile stress. Quantitatively, *k* had an average value of about 70% under both stresses at the initial stress level, and the values of *k* were about 30% and about 45% for the uniaxial compressive stress and tensile stress, respectively, at a stress level of around 35%. The relative deformation of the crystalline region decreased under both stresses, and was especially remarkable under uniaxial compressive stress.
2) In the stress range beyond the stress level of about 35%, *k* increased to a stress level of about 45% under uniaxial tensile stress, after which its value was almost constant. Under uniaxial compressive stress, *k* could not be measured after a stress level of about 35% owing to measurement problems. The piezoelectric voltage determined using a different measurement system was plotted to predict the behavior of *k* in the stress range. It is thought that the fluctuation behavior of *k* appears to be predictable from that of the piezoelectric voltage. In short, in the stress level range from about 35% to failure under uniaxial compressive stress, from the piezoelectric voltage behavior it can be predicted that *k* increases up to a stress level of about 45%, after which it declines in the form of a curve.
3) It is anticipated that after a stress level of about 45%, the relative deformation of the crystalline region differs under the two types of stress.

Key words: Wood, Cellulose crystal lattice strain, Crystalline region, Macroscopic surface strain, Uniaxial stress in the fiber direction, (004) reflection, Transit X-ray method

1. Introduction

Wood, a composite material, is composed mainly of natural cellulose and matrix. The natural cellulose that forms the skeleton comprises about half of the components (crystallinity: about 50-60%). Although it is probable that natural cellulose

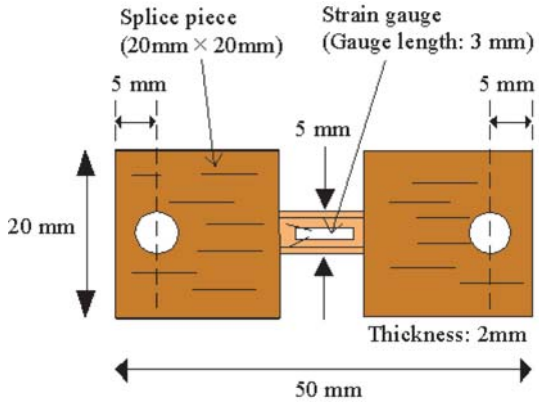
greatly affects the dynamic behavior of wood, there have been few reports[1-3] on the concrete effects of the crystalline and non-crystalline regions in wood on the macroscopic and semi-micro dynamic behaviors in small clear specimens. In the present study, X-ray stress measurements were performed by applying uniaxial compressive stress and tensile stress to test specimens in the direction of the fibers. The relationship between the crystal lattice strain and the macroscopic strain in the test specimens (hereafter referred to as the surface strain) was investigated in detail. Bragg's equation was used to calculate the crystal lattice strain. The experiment was carried out taking into account the microfibril inclination angle (MFA, by Cave's method) of the test piece, as it is thought that the orientation of the fibers greatly affects the dynamic behavior.

2. Materials and Methods

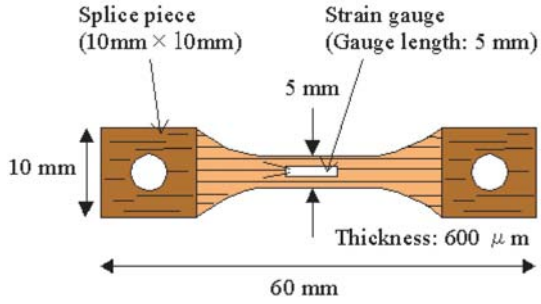
Specimens studied The crystal lattice strain and piezoelectric voltage were measured in five specimens of Japanese cypress (*Chamaecyparis obtusa* Endl.), which were first conditioned at a constant temperature of 20ºC and a constant humidity of 60 % for 6 months. Before testing, the specimens had the following characteristics: density 0.33 ± 0.01 g/cm^3 (mean ± S.D.), annual ring width 1.5 ± 0.02 mm, percent late wood 23.8 ± 0.15 %, microfibril angle 12.0º-16.8º, crystallinity 54 ± 1.5%, and moisture content 11.9 ± 0.1%.

Straight-grained pieces of wood were used to measure the X-ray stress. The pieces used for the compressive tests had external dimensions of 2 mm × 2 cm × 50 mm (thickness width length) and those used for the tensile tests had dimensions of 600 μm × 10 mm × 60 mm. Arches were cut in both sides of the center of each test specimen to form a constriction, and beech wood was glued to the specimens with epoxy resin as a reinforcement. A 5-mm-diameter hole was drilled in each test specimen to fix it to the jig and thus prevent slippage. To measure the macroscopic surface strain of the test specimens, a 5-mm-long strain gauge (Tokyo Sokki Kenkyujo Co., Ltd., Japan) was glued to the center of the specimen using cyanoacrylate adhesive. The effects of the strain gauge and adhesive on the strength of the test specimen were examined by varying the thickness of the test specimen. They had significant effects when the thickness was less than 500 μm; whereas the intensity was constant when the thickness exceeded 600 μm. Consequently, test specimens greater than 600 μm thick were used for the X-ray stress measurement and tensile testing.

In addition, test specimens with external dimensions of 9 cm × 3 cm × 3 cm (height × width × thickness) were prepared to measure the piezoelectric voltage. We attached plain electrodes to a radial section such that the angle between the axial direction and fiber direction of the specimen was 0 degrees. The electrodes used to detect the piezoelectric voltage were 3 cm × 3 cm × 100 μm pieces of aluminum foil (length × width × thickness) bonded to the center of opposite sides of the specimen with electrically conductive double-sided tape. The lead wires were bonded to the electrodes using electrically conductive paint.



(a) Specimen used for uniaxial compressive test.



(b) Specimen used for uniaxial tensile test.

Fig.1. Schematic diagram of the test specimen used for X-ray stress measurement.

X-ray stress measurement Natural cellulose, the main constituent of wood, crystallizes in the cell wall to form rigid microfibrils. The microfibrils are aligned roughly parallel to the direction of the fibers in the middle layer of the secondary wall, which constitutes the majority of the cell wall. The change in the spacing of the crystal lattice caused by stress loading in the fiber direction (*i.e.*, the crystal lattice strain) can be measured using X-ray diffraction[1-3]. To determine the strain in the loading direction in a material such as wood, which contains both aligned crystals and irregular non-crystalline regions, direct measurements using a transmission method are more effective than measurements using a reflection method. Therefore, the intensity of the (004) diffraction peak was measured using a transmission method to determine the change in the crystal lattice strain in test specimens of wood under uniaxial stress in

the fiber direction.

A jig constructed for this experiment was attached to the rotating part of the sample holder on a goniometer (Fig. 2). Each test specimen was attached to the jig with screws, and a load was placed on it *via* a bar for compressive tests or a wire for tensile tests under the control of a small desktop material-testing machine. An X-ray diffractometer (XD-D1w, Shimadzu Co., Japan) was used to measure the crystal lattice strain under the following conditions: Cu X-ray tube target; CuKα characteristic X-ray line (Ni-filter correction); tube voltage, 40 kV; tube current, 40 mA; air scattering prevention slit angle, 1°; divergence slit angle, 1°; detection slit width, 0.1 mm; continuous scan mode; drive shaft, -2 ; integration time, 4 sec; and scan speed, 0.125°/min. In addition, the diffraction intensity profile was measured around the diffraction peak of the (004) plane (2 = 32°-37°), and the diffraction angle was approximated using an asymmetric Gaussian curve. After attaching the specimen to the tension jig, the diffraction intensity profile was first measured with no load, and then measured for a constant interval until the specimen failed. When a specimen is loaded with a constant stress, the stress actually decreases owing to stress relaxation. To prevent this decrease, the X-ray stress measurement was measured while monitoring the stress on a personal computer so that the stress could be maintained manually. With the test specimen fixed to the jig, loading was carried out after measuring the diffraction intensity profile under the no-load condition. Next, the diffraction intensity profile was measured at each step up to the break load value.

The crystal lattice strain was calculated using Bragg's equation:

$$n\lambda = 2\bar{d}\sin\theta, \quad \cdots (1)$$

where n is the degree of reflection; λ is the X-ray wavelength; $\bar{d}$ is the lattice spacing; and is the Bragg angle. If the lattice spacing changes by $\Delta\bar{d}$ when under a load, and the diffraction peak of the characteristic X-ray changes by Δ proportionally, then Equation (1) can be rewritten by replacing $\bar{d}$ and with $\bar{d}+\Delta\bar{d}$ and $+\Delta$, respectively, as:

$$n\lambda = 2(\bar{d}+\Delta\bar{d})\sin(\theta+\Delta\theta), \quad \cdots (2)$$

This gives:

$$n\lambda = 2(\bar{d}+\Delta\bar{d})\left\{\sin\theta+\cos\theta\,\Delta\theta-\frac{1}{2}\sin\theta(\Delta\theta)^2+\cdots\right\}, \quad \cdots (3)$$

Here, if $(\Delta)^2$ and $\Delta\ \Delta d$ are disregarded as negligible, Equation (3) can be rewritten as:

$$n\lambda = 2\bar{d}\sin\theta+2\bar{d}\cos\theta\,\Delta\theta+2\sin\theta\,\Delta\bar{d}, \quad \cdots (4)$$

From Equations (1) and (4), we obtain:

$$\frac{\Delta\bar{d}}{\bar{d}} = -\cot\theta\cdot\Delta\theta, \quad \cdots (5)$$

Therefore, the amount of crystal lattice strain can be calculated from Equation (5) using the difference in the peak value of the diffracted intensity profile in the unloaded and loaded states, Δ .

(a) The jig used to apply compressive stress to the specimen.

(b) The jig installed in the X-ray diffraction equipment.

Fig.2. Apparatus used to measure X-ray stress.

Piezoelectric voltage detection and loading method The piezoelectric voltage generated by the wood was transmitted *via* the electrodes and amplified by a1/3-octave band pass filter with an input impedance of 10 MΩ (NEC Sanei Co., Ltd.) to remove noise. The voltage was then measured with a highly sensitive alternating current voltmeter with a built-in AC-DC converter (NF Circuit Design Block Co., Ltd.). In this study, the measured piezoelectric voltage had an extremely high signal-to-noise ratio.

The load was applied with a testing machine using oil pressure (servo pulser, EHF-UG100kN-20L, full-scale range = ±100 kN, Shimadzu Co., Ltd.). The specimens were attached to the jig, and static tension with a minute superimposed sinusoidal load (F), given by Equation (6) below, was applied. The order of loading was as follows: an initial load of F_1 = 0.25 kN was applied, then a sinusoidal load with a frequency of f = 30 Hz and an amplitude of a = 0.2 kN was applied for 10 seconds (t). Load F_n was increased at a constant interval until the specimen failed. The sinusoidal load made it

possible to detect the piezoelectric voltage using this apparatus. The combined load, F, is given by:

$$F = F_n + a \cdot \sin((2\lambda f)t), \quad \cdots (6)$$

Measuring systems The crystal lattice strain (Fig. 3) and piezoelectric voltage were measured in a room kept at a constant temperature and humidity of 20ºC and 60%, respectively. For the X-ray stress analysis, the output of a load cell built into the jig and the surface strain on the specimen were collected simultaneously at 100-ms intervals using a data acquision controller (DE-1200IF, NEC San-ei Co., Ltd.), which also recorded the piezoelectric voltage (load, displacement). The data were stored in a personal computer.

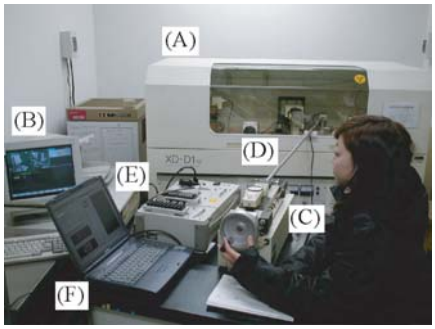


Fig. 3. System used for the X-ray stress analysis.
Legend: (A) X-ray diffraction equipment, (B) Personal computer for controlling (A), (C) Loading device (material-testing machine), (D) Bar for applying compressive load to jig, (E) Data acquisition controller, (F) Personal computer for storing the experimental data.

4. Results and Discussion

The ratio of the crystal lattice strain to the surface strain, k, was plotted for the stress applied to the test specimens (Fig. 1).

Up to a stress level of about 35%, the dynamic behavior of the crystalline region in the wood showed very similar qualitative tendencies under uniaxial compressive stress and uniaxial tensile stress. That is, k changed from an increase to a decrease with increased stress. At the same time, quantitatively, k had an average value of about 70% under both stresses at the initial stress level, and the values of k were about 30% and about 45% for the uniaxial compressive stress and tensile stress, respectively, at a stress level of around 35%. The relative deformation of the crystalline region decreased under both stresses, and was especially remarkable under uniaxial compressive stress.

In the stress range beyond the stress level of about 35%, k increased to a stress level of about 45% under uniaxial tensile stress, after which its value was almost constant. Under uniaxial compressive stress, k could not be measured after a stress level of about 35% owing to measurement problems.

The piezoelectric voltage[4-7] determined using a different measurement system was plotted to predict the behavior of k in

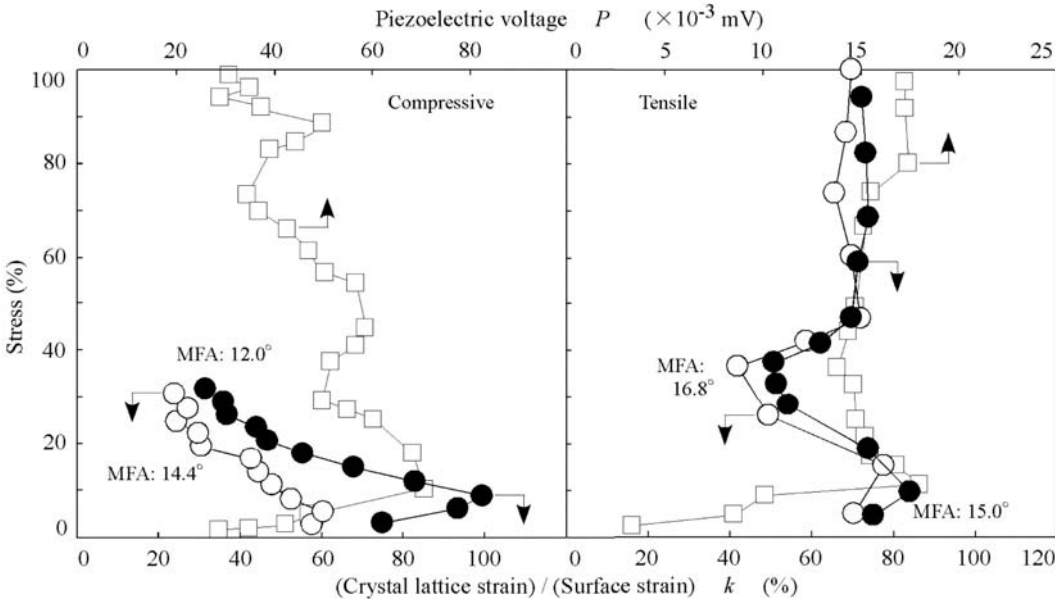


Fig. 4. Relationships between the ratios of the crystal lattice strain to the surface strain, the piezoelectric voltage, and the stresses.
Legend: *Arrows* show the horizontal axis of each curve, MFA: Microfibril angle.
Note: The stress values depicted in the vertical lines are given as the percentage of the breaking stress.

the stress range. As shown in the figure, both correspondences were good in all stress ranges under uniaxial tensile stress. It is thought that the fluctuation behavior of k appears to be predictable from that of the piezoelectric voltage. In short, in the stress level range from about 35% to failure under uniaxial compressive stress, from the piezoelectric voltage behavior it can be predicted that k increases up to a stress level of about 45%, after which it declines in the form of a curve.

It is anticipated that after a stress level of about 45%, the relative deformation of the crystalline region differs under the two types of stress. In wood, it is well known that the tensile strength is bigger than the compressive one. We estimated that this experimental fact is a cause on the difference between each strength.

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