LAAN-A-SA-E006

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Application News

No. S32

Surface Observation

Morphological Observation of Cellulose Nanofiber Obtained by Mechanical Defibration

Introduction

Cellulose is a polysaccharide consisting mainly of plant cell walls. Nanocellulose is produced by defibrating cellulose to the nanometer size. Nanocellulose with a width of 4 to 100 nm, length of approximately several μm, and high aspect ratio (100 or more) is called cellulose nanofiber (CNF), and is a focus of attention as an advanced new biomass material.

In addition to light weight and high strength, CNF also offers outstanding functions such as a high gas barrier property, adsorption, and transparency. Moreover, because CNF is a plant fiber-derived material, the environmental impacts associated with production and disposal are small. Application to automotive, electronic, packaging and other materials is expected in the future.

Because CNF is produced by the grinder method, in which cellulose is mechanically defibrated, optimization of the defibration conditions is required for efficient production of larger quantities of CNF. Therefore, morphological observation was performed focusing on the fibers in the defibration process, which had not been considered important in the past. This article introduces an example of morphological observation of fibers during the mechanical defibration process using a scanning probe microscope (SPM) and laser scanning microscope (LSM).

E. Iida

Instruments Used in Observation

SPM enables high magnification observation of the three-dimensional (3D) profile and local physical properties of samples by scanning the sample surface with a microscopic probe (observation range: several tens nm to 125 μm). LSM enables noncontact, wide-range observation of the 3D profiles of samples (observation range: 16 to 2560 μm). Fig. 1 shows the appearance of the instruments.

Fig. 1 OLS Series 3D Measuring Laser Microscope (left), SPM-9700HT™ Scanning Probe Microscope (right)

Morphological Observation of Fibers During Defibration

Samples were prepared by defibrating a broadleaf tree pulp sheet a maximum of five times by the grinder method, followed by dispersion in water. Because the concentration and viscosity of the samples are high in the defibrated state, the fibers were diluted 400× with water after each defibration treatment, and were observed after dripping/drying on a cleaved mica surface. Due to the large size of the untreated fibers and fibers after one treatment, these fibers were observed by LSM (observation field: 260 μm × 260 μm). Fibers after two to five treatments were observed by SPM, and the average fiber diameters were calculated (observation field: 10 μm × 10 μm). Fig. 2 shows the observation results. Although the untreated fibers are in a bundled condition, it can be seen that the fibers unravel and become progressively finer as defibration treatment proceeds. Since the decrease in the average fiber diameter from the 2nd treatment to the 3rd treatment was particularly large, it is thought that the largest defibration occurs in this process.

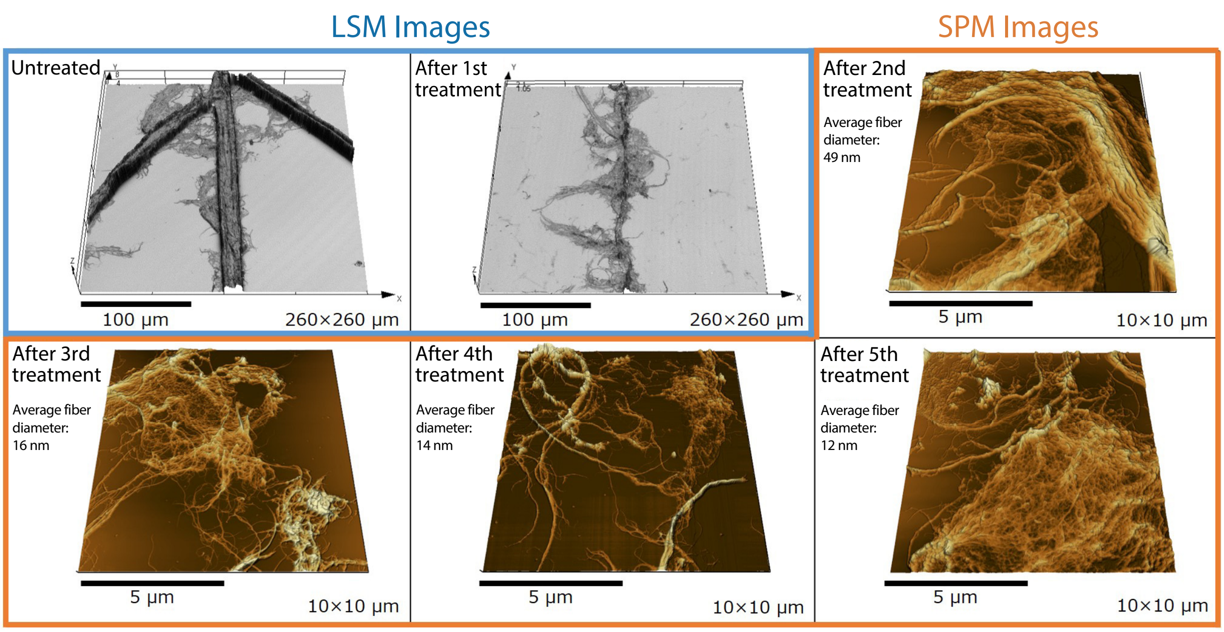

Fig. 2 Images of Fiber Shape and Average Fiber Diameter After Each Stage of Defibration Treatment

High Resolution Observation of CNF

The fibers obtained by performing defibration treatment five times (Fig. 2, lower right) were observed with high resolution by SPM (observation field: 250 nm × 250 nm). Fig. 3 shows the result (high resolution observation region: area outlined in red in figure at left). The shape of the CNF can be seen clearly in Fig. 3.

The cross-sectional profile was analyzed in order to measure the fiber diameter of the CNF. With SPM, the height value is generally used as the fiber diameter because the sample profile width is observed as larger than the actual size due to effect of the probe shape. From Fig. 4, it can be understood that the fiber diameter of the CNF is on the order of 4 nm (red box in Fig. 4). This demonstrates that individual CNF fibers can be captured by high resolution observation.

Conclusion

The morphological changes in CNF due to mechanical defibration treatment were observed by LSM and SPM. High resolution observation of individual CNF fibers was realized by SPM. In the future, progress in optimizing CNF defibration conditions is expected to contribute to more efficient production of CNF.

<Acknowledgments>
We wish to express our gratitude to Assistant Professor Akihiro Hideno of the Paper Industry Innovation Center of Ehime-university (PIICE) for providing and evaluating the CNF samples and his generous guidance.

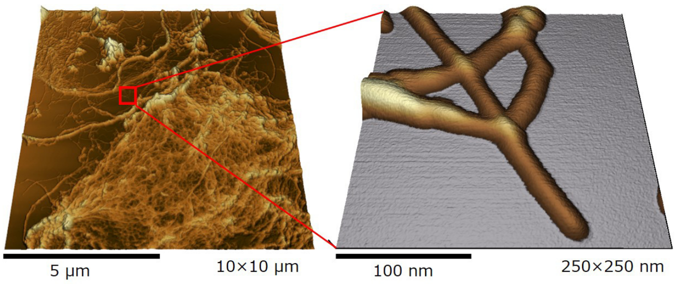

Fig. 3 High Resolution Observation of CNF After 5th Defibration Treatment

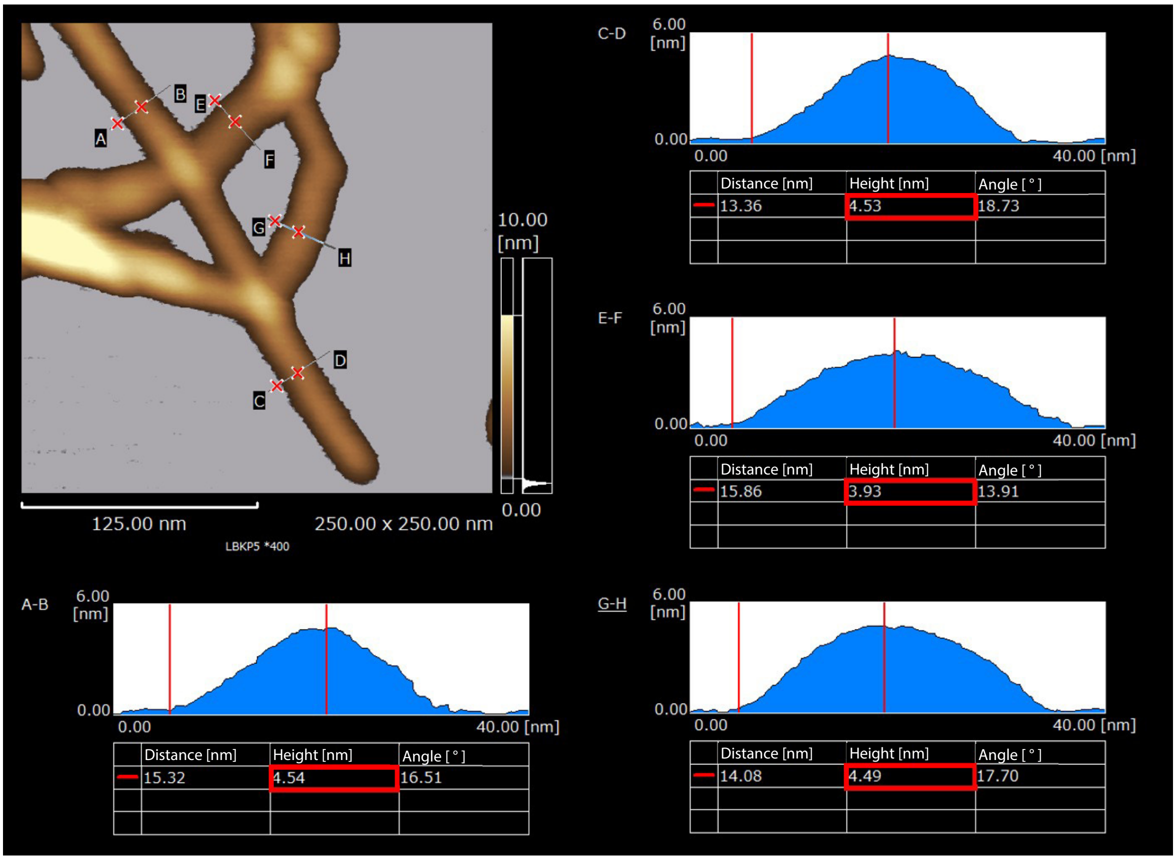

Fig. 4 Cross-Sectional Profile Analysis of Individual CNF Fiber

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First Edition: Sep. 2018

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