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Application News

SPM-Nanoa™/SPM-9700HT™ Scanning Probe Microscope/Atomic Force Microscope
OLS5100 3D Measuring Laser Microscope

Observations of the Shapes of a Variety of Nanofibers

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User Benefits

- From nanometer scale to micrometer scale, we can observe the shapes of nanofibers made from different materials and different manufacturing methods.
- The fine structure of a single nanofiber can be clearly observed.
- The fiber diameter of individual nanofibers can be measured.

■ Introduction

Nanofibers such as cellulose nanofibers (CNF) have diameters in the order of a few nm to 100 nm. In combination with plastics, they form composite materials with potential applications in the fields of automotive parts, electronic materials, and packaging materials. The shapes and sizes of nanofibers are largely responsible for the mechanical strength of these composite materials. Accordingly, assessing and controlling these attributes are important for developing and manufacturing composite materials suited for particular applications. The shapes and sizes of nanofibers vary depending on the raw material and manufacturing method, and in the manufacturing process, micrometer-sized nanofibers are not unusual. Combining a laser scanning microscope (LSM) with a scanning probe microscope (SPM) /atomic force microscope (AFM) makes it possible to check the shapes and sizes of everything from fibers in the manufacturing process (micrometer-sized fibers) to finished products (nanometer-sized fibers).

This article introduces examples of the use of an LSM and SPM to assess the shapes and sizes of various nanofibers with different raw materials and manufacturing methods.

■ Observing the Shapes of Various Nanofibers

The samples observed were six types of nanofibers made from different raw materials and using different manufacturing methods (Fig. 1). An LSM was used for the overall assessment of (a) grinder defibered CNF and (b) fermented nano cellulose, samples with prominent aggregation of fibers. The LSM measurement conditions are shown in Table 2. To assess the individual fibers, the SPM was used to observe all six types. The SPM measurement conditions are shown in Table 3. For (a) grinder defibered CNF and (b) fermented nano cellulose, in which aggregation of fibers is particularly well established, LSM was used at first to find suitable observation positions for SPM.

Table 1 Measurement Samples

	Sample Name	Raw Material	Manufacturing Method
(a)	Grinder Defibered CNF	Cellulose	Grinder
(b)	Fermented Nano Cellulose	Acetobacterium	Fermentation
(c)	Water Jet Defibered CNF	Cellulose	Water Jet
(d)	Chitin Nanofibers	Chitin	Water Jet
(e)	Carboxymethyl Cellulose (CMC)	Cellulose	Chemical Treatment
(f)	TEMPO-Oxidized CNF	Cellulose	TEMPO Oxidation Treatment

Table 2 LSM Measurement Conditions

Instrument:	OLS5100 3D Measuring Laser Microscope (OLYMPUS Corp.)
Objective Lens:	Dedicated OLS 100× Objective Lens
Observation Field of View:	130 µm × 130 µm

Table 3 SPM Measurement Conditions

Instrument:	SPM-Nanoa/SPM-9700HT Scanning Probe Microscope
Scanner:	HT Scanner (XY: 10 µm)
	Wide Area Scanner (XY: 125 µm)
Measurement Mode:	Dynamic Mode
Observation Field of View:	2.5 µm × 2.5 µm, 250 nm × 250 nm

Fig. 1 shows height images from the LSM observations. The colors are artificial. In (a), with the grinder defibered CNF, narrow fibers are aggregated in the vicinity of the thick, straight fibers. In (b) with the fermented nano cellulose, it is evident that fibers with the same thickness are aggregated at a size of approximately 100 µm at maximum.

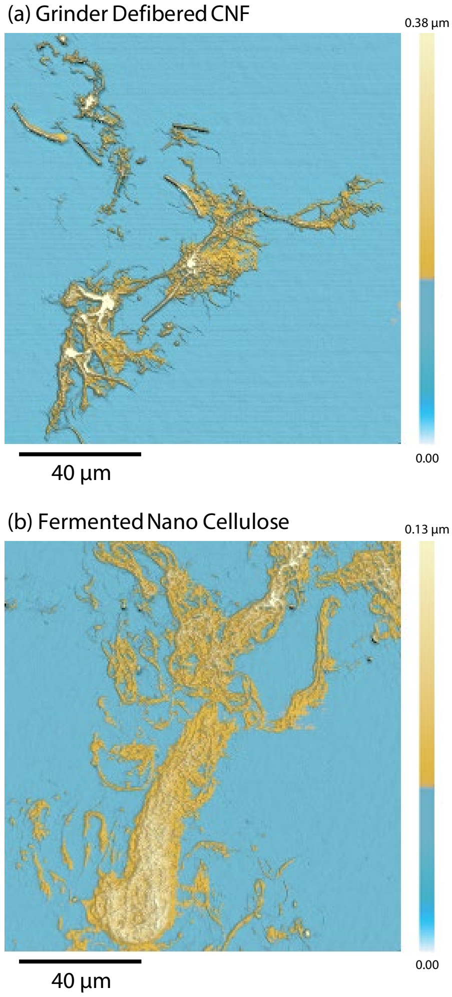

Fig. 1 LSM Height Images

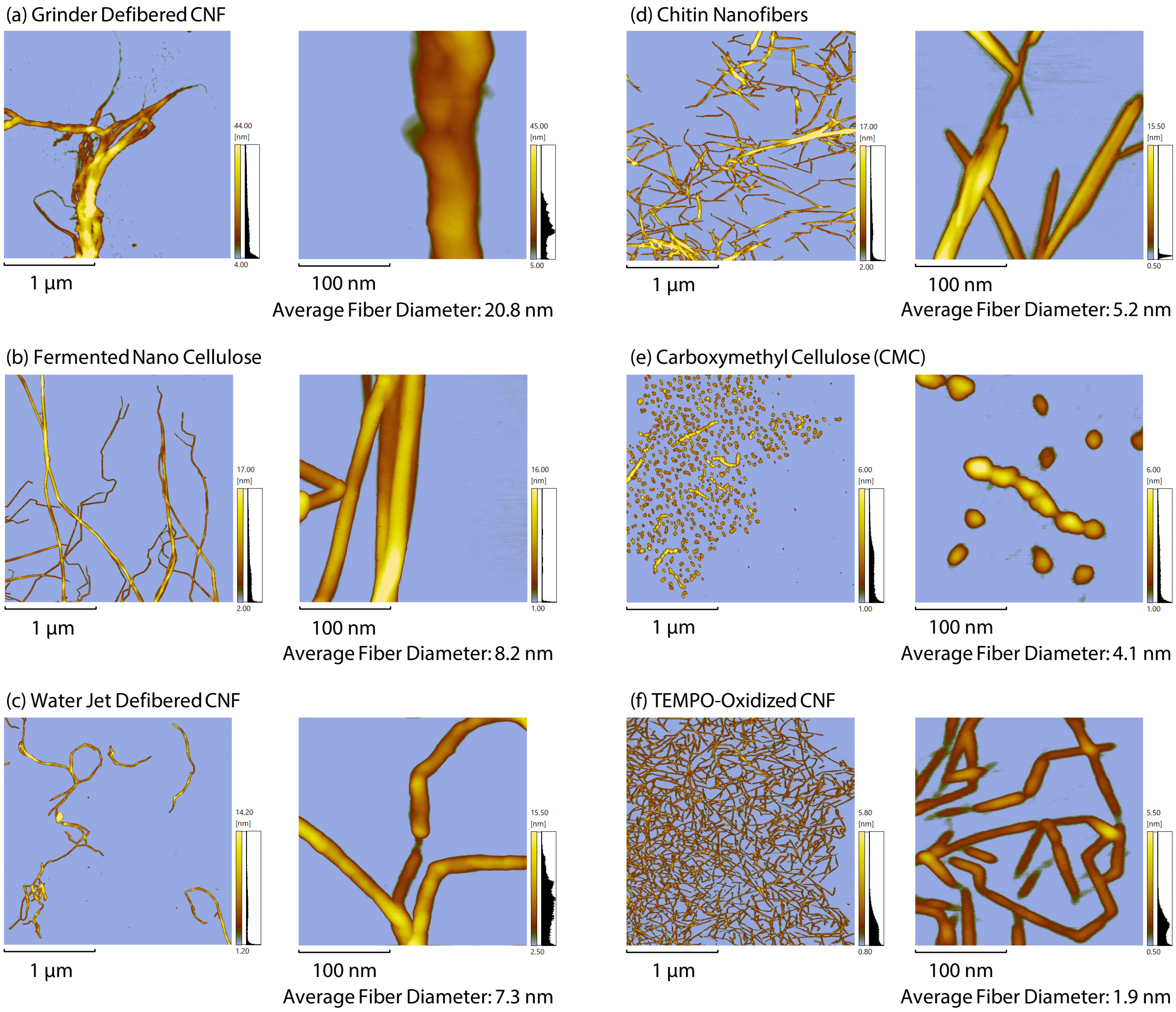

Fig. 2 SPM Height Images

Fig. 2 shows height images from the SPM observations. The colors are artificial. At five arbitrary positions within the SPM observation image enlarged to 250 nm x 250 nm, the average fiber diameter was calculated by measuring the fiber height from the cross-sectional shape profiles as the fiber diameter.

The results for (a) grinder defibered CNF show a large fiber diameter, with fiber branching and crossovers. The (b) fermented nano cellulose fibers are long, and many of the fibers are straight. In the (c) water jet defibered CNF, finer defibering is achieved than with (a) grinder defibering, another mechanical defibering method. The fibers with smaller diameter are isolated and dispersed as single fibers. The (d) chitin nanofibers are short, and many are straight. The (e) CMC and (f) TEMPO-oxidized CNF fibers are small in diameter, and are isolated and dispersed as single fibers. It is thus apparent that the shapes, lengths, and degrees of dispersion differ depending on the raw material and the manufacturing method.

■ Conclusion

The shapes of various nanofibers produced with different raw materials and manufacturing methods were observed using an LSM and SPM. At the initial stage of manufacturing, samples contain many micrometer-sized fibers, which are ideal for LSM observations. In contrast, SPM is optimal for measuring fine, easily dispersed nanometer-sized fibers. Using an LSM and SPM in combination to suit the shapes and sizes of the fibers is an effective technique.

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