SHIMADZU
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Application News

No. S30

Surface Observation

Observation of Cellulose Nanofibers and Measurement of Fiber Length/Width

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.

The lack of an established method for evaluating the basic physical properties of CNF is one current issue. As basic measurements, establishment of a method for measuring the fiber length and width of CNF is demanded, as they are thought to influence the mechanical strength of CNF composites. The scanning probe microscope (SPM) and electron microscope are generally used to observe nanometer-size objects, and are also widely used in observation of CNF.

This article introduces a method for observation of CNF and measurement of the fiber length/width by using Shimadzu's scanning probe microscope SPM-9700HT™.

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Scanning Probe Microscope SPM-9700HT

The SPM enables high-magnification observation of the 3-dimensional shape and local properties of samples by scanning the sample surface with a microscopic probe. The appearance of the SPM-9700HT and the principle of observation are shown in Fig. 1 and 2, respectively.

Fig. 1 Scanning Probe Microscope SPM-9700HT™

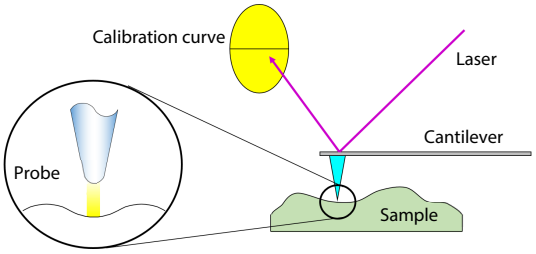


Fig. 2 Principle of SPM

Shape Observation of CNF

The samples measured here are commercial water-dispersed cellulose products.*1 Five types with the different fiber lengths (extra-long, long, standard, short, extra-short) shown in Fig. 3 were observed. The samples were adjusted to a concentration of 0.001 wt%, dropped on a cleaved mica surface, dried, and then observed. Fig. 4 shows the shape images of a 10 µm × 10 µm observation field, which makes it possible to grasp the total image. Fig. 5 shows the shape images of a 2.5 µm × 2.5 µm field enlarged for evaluation of the fiber length.

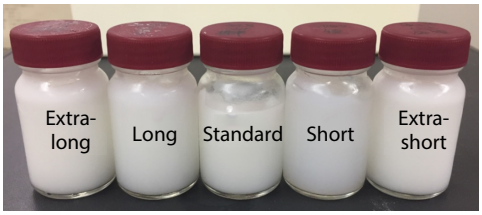


Fig. 3 Water-Dispersed Cellulose

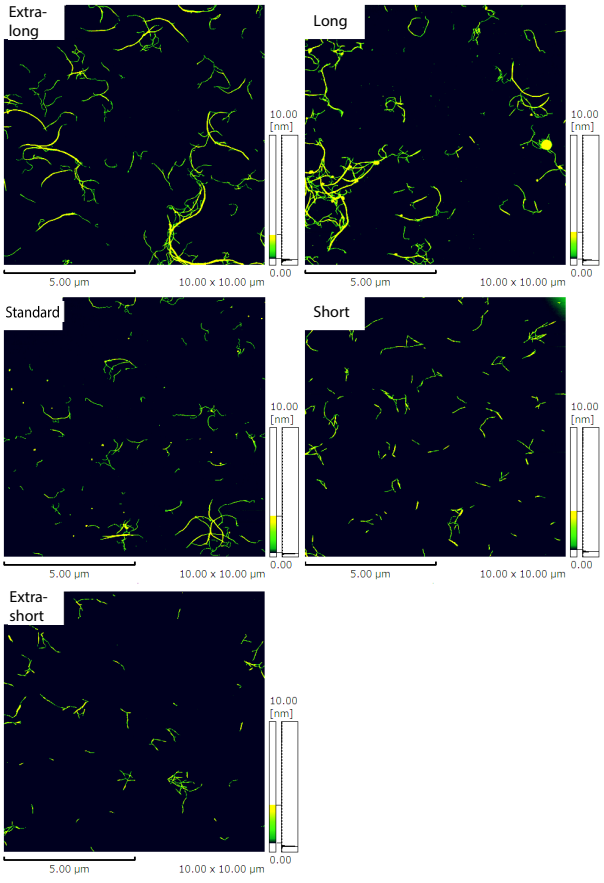


Fig. 4 Shape Images of CNF (Observation Field: 10 µm × 10 µm)

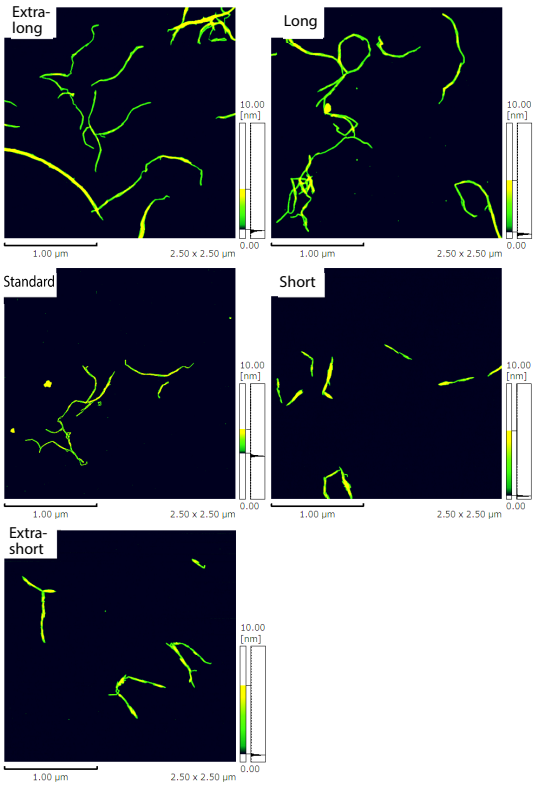


Fig. 5 Shape Images of CNF (Observation Field: 2.5 µm × 2.5 µm)

CNF Fiber Length/Width Measurement Method

Particle analysis software was used in measurements of the fiber length and width. The software first extracts the contours of the CNF as particles from the obtained 3D shape images, and then calculates the feature values of multiple extracted particles, enabling statistical analysis. The dedicated particle analysis software*2 of SPM-9700HT supports 29 types of feature values, including length and height.

In fiber extraction, as shown in Fig. 6, a threshold is set based the height information from the obtained images, and part of the image that contains a large portion of fibers is used as the extraction object.

Because CNF has a high aspect ratio and a negligibly small width-to-length ratio, the fiber length is calculated as "circumference/2" and the fiber width is calculated as the average of Z (height) using the feature values after extraction.

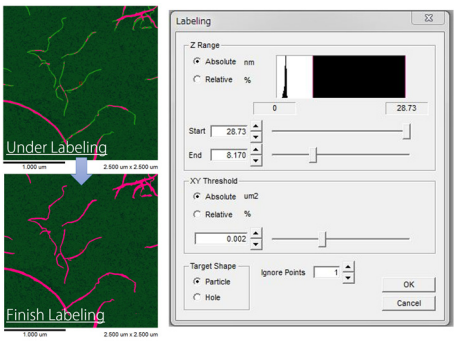
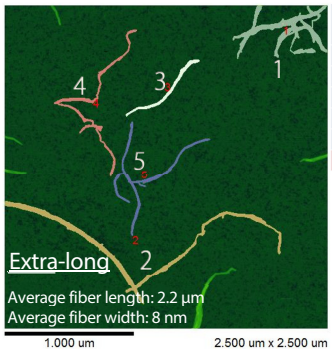


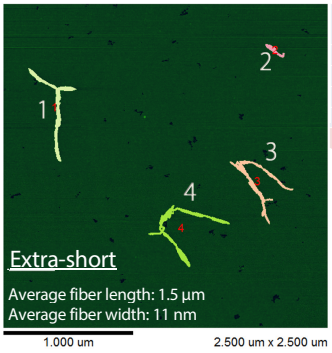
Fig. 6 Particle Extraction Process of Particle Analysis Software

Fig. 7 and 8 show the measured results of "Extra-long" and "Extra-short," respectively. The average fiber length and width of "Extra-long" were calculated as 2.2 µm (from 4.3/2) and 8 nm, and the values for "Extra-short" were calculated as 1.5 µm (2.9/2) and 11 nm. The "Extra-long" fibers included some that did not fit completely within the image, and in the case of fibers that appeared consisting of several intertwined fibers, the entire fiber was counted as one fiber. The values of fiber length and width differ greatly depending on the degree of defibration which is regarded as one fiber. Thus, clarification of the condition regarded as one fiber is an issue, particularly when measuring the fiber length.



No.	Circumference [µm]	Average of Z [nm]
1	4.6	10
2	6.5	10
3	1.6	6
4	4.3	6
5	4.4	6
Average	4.3	8

Fig. 7 Measured Results of "Extra-Long" CNF



No.	Circumference [µm]	Average of Z [nm]
1	2.1	12
2	4.3	9
3	2.5	10
4	2.5	11
Average	2.9	11

Fig. 8 Measured Results of "Extra-Short" CNF

Conclusion

Observation of CNF and measurement of the fiber length/width can be performed with an SPM with sub-nanometer resolution. Easy calculation of the fiber length/width is possible by applying particle analysis software to the 3-dimensional shape data. In the future, clarification of the definition of "one fiber" will be important for accurate measurement.

*1 Sugino Machine Limited, BiNFi-s® water-dispersed cellulose
Product Nos.: IMa-10002 (Extra-long), BMa-10002 (Long), WMa-10002 (Standard), AMa-10002 (Short), FMa-10002 (Extra-short)

*2 C147-3093 SPM-9700HT catalog, p. 15
Particle Analysis Software

SPM Data Room
SPM Data Room introduces various applications of SPM.
https://www.shimadzu.com/an/surface/spm/dataroom.html

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