SHIMADZU
Excellence in Science

Application News

No. S39

Surface Observation

Automatic Shape Observation and Efficient Length Measurement of Fiber Length/Diameter of Cellulose Nanofibers

Introduction

Cellulose is a polysaccharide which is the main component of plant cell walls. Among nanocellulose obtained by defibrating cellulose to the nanometer size, fibers with a diameter of several nm to around 100 nm are called cellulose nanofiber (CNF). CNF has attracted attention as a leading-edge biomass new material. In addition to offering a combination of light weight and high strength, CNF also has other outstanding functions, such as a high gas barrier property, adsorption property, and transparency. Moreover, because CNF is derived from plant fiber, environmental loads related to production and disposal are minimal. Future applications are expected to include automotive components, electronic materials, and packaging materials, among others.

Because the fiber length and diameter of CNF are thought to influence the physical properties of CNF composites, there is a heightened need for characterization of single CNF fibers in development and manufacturing. However, improvement of characterization efficiency has become an issue, as shape observation and length/diameter measurement of several thousand individual fibers are necessary in characterization of CNF. This article introduces an example of automatic shape observation/length measurement of CNF by using a scanning probe microscope (SPM).

E. Iida

Instrument Used in Observation

The SPM enables high-magnification observation of the 3-dimensional shape and local physical properties of samples by scanning the sample surface with a microscopic probe. Fig. 1 shows the appearance of the SPM used in this experiment. Dedicated particle analysis software for the SPM-9700HT™ was used in the length measurements of the fiber length/diameter of the observed CNF.

Fig. 1 SPM-9700HT™ Scanning Probe Microscope

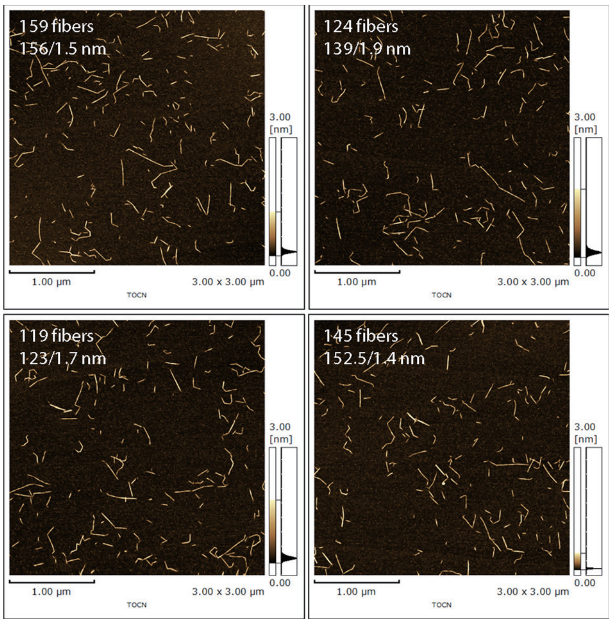

Fig. 2 Results of Automatic Shape Observation of TOCN (TEMPO-Oxidized CNF) (Numbers in Images: Number of TOCN and Average Fiber Length/Average Fiber Diameter)

Automatic Shape Observation of TEMPO-Oxidized CNF

The CNF observed here was TEMPO-oxidized CNF (TOCN). TOCN is defibrated to the nanometer size by a chemical reaction called TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl)-mediated oxidation and a gentle mechanical processing. TOCN has a uniform fiber diameter of 2 to 3 nm. Since it has the features of high transparency and dispersibility in solutions, application to composite materials with resins and rubber and to paints is expected.

TOCN dispersed in water was diluted to 0.001 wt% with water, dripped on a cleaved mica surface and dried, and automatic observation of 16 fields of view was carried out. In automatic observation, continuous observation of multiple fields by pushing one start button was possible by setting the observation field size, scanning speed, and offset movement amount of the observation field in advance. To prevent observation of the same fiber, a space of 3 µm was left between adjacent fields. Fig. 2 shows typical observation results. The observation field size is 3 µm × 3 µm, and the height scale is 3 nm. The individual TOCN can be seen clearly, and a condition of moderate dispersion can be observed.

Length Measurement of Fiber Length/Diameter

Length measurement of half of the perimeter of the fibers as the fiber length and the average height of the fibers as the fiber diameter was conducted by extracting the TOCN as particles by applying the particle analysis software to each image of the 16 fields of view. For accurate length measurement, TOCN that was not completely included in an observation field of view was excluded. Fig. 3 and Fig. 4 show the results of the length and diameter measurements, respectively. Here, 2307 TOCN could be extracted. Their average fiber length was 144.6 nm and average fiber diameter was 1.7 nm. Regarding the fiber length, approximately half of the TOCN were distributed in the range of 30 nm to 150 nm, and some TOCN with lengths of 300 nm or more were also observed. The diameters of almost all of the TOCN were distributed in the range of 1 nm to 3 nm, centering on 1.7 nm. The fiber diameter was extremely uniform, and consistency with the physical property value (2 to 3 nm) was good.

Thus, it was possible to obtain a detailed knowledge of the distributions of the fiber length and fiber diameter from the actually observed shape images.

Conclusion

Automatic shape observation of CNF and length measurement of the fiber length/diameter were realized by using SPM. Although manual measurement of 200 to 300 CNF fibers requires several hours, about 2300 fibers could be measured in almost the same time in this experiment. Because this method is applicable not only to CNF, but also to fine particles like nanoparticles, wide application as a technique that dramatically improves efficiency in shape and particle diameter characterization is expected.

<Acknowledgments>
We wish to thank Prof. Akira Isogai, Associate Prof. Tsuguyuki Saito, and Research Associate Shuji Fujisawa of the University of Tokyo for providing the CNF samples and valuable guidance in the characterization.

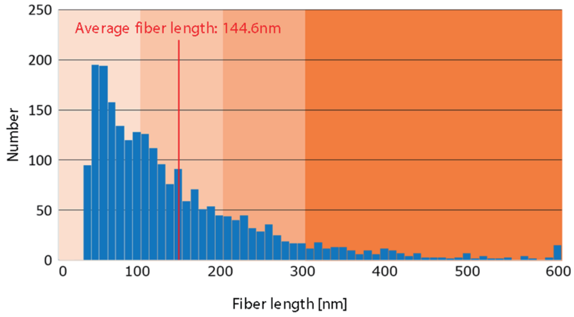

Fig. 3 Distribution of TOCN Fiber Length (Lengths over 600 nm: Shown as 600 nm)

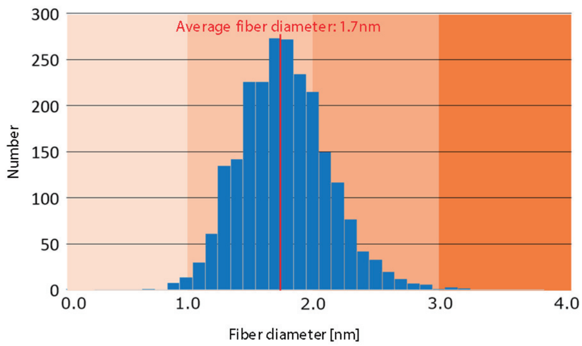

Fig. 4 Distribution of TOCN Fiber Diameter

SPM-9700HT is a trademark of Shimadzu Corporation in Japan and/or other countries.

First Edition: Jul. 2019

Shimadzu Corporation
www.shimadzu.com/an/

For Research Use Only. Not for use in diagnostic procedures.
This publication may contain references to products that are not available in your country. Please contact us to check the availability of these products in your country.

The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu. Shimadzu disclaims any proprietary interest in trademarks and trade names used in this publication other than its own. See http://www.shimadzu.com/about/trademarks/index.html for details.

The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

© Shimadzu Corporation, 2019