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Excellence in Science

Application News

No. S37

Surface Observation

Observation and Characterization of Dispersion State of Cellulose Nanofibers and Polymer Composite

Introduction

Cellulose is a polysaccharide, which is a main component of plant cell walls. Among nanocellulose materials produced by defibrating cellulose to the nanometer size, those with a width of 4 to 100 nm, length on the order of several µm, and high aspect ratio (100 or higher) are called cellulose nanofibers (CNF), and have attracted attention as a leading-edge new biomass material. Because CNF has a variety of outstanding functions, including a high gas barrier property, absorption, and transparency, in addition to light weight and high strength, composites of CNF with polymer materials are expected to display improved physical properties.

Although characterization of the dispersion states of CNF and polymer composite in the material is important in the development of composite materials, the current characterization techniques cannot be considered adequate.

This report introduces an example of a CNF composite material and characterization of its dispersion state by using a Shimadzu scanning probe microscope (SPM) and Nano Search microscope (SFT).

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Observation Instruments

SPM applies a technique in which the surface of a sample material is scanned with a microscopic probe, enabling high-magnification observation of the 3-dimensional topography and local physical properties of the sample. Nanophysics Evaluation System "Nano 3D Mapping™ " was used in characterization of the dispersion state. SFT is a hybrid instrument consisting of a laser scanning microscope (LSM) and SPM, and makes it possible to observe the 3-dimensional topographies of sample materials over a wide range of magnifications. Fig. 1 shows the appearance of these instruments.

Fig. 1 Nano Search Microscope SFT-4500 (Left), Scanning Probe Microscope SPM-9700HT™ (Right)

Wide-Range Observation of CNF/PVP Composite Material

The sample material was prepared by mixing a CNF aqueous solution (1 wt%) and a polyvinyl pyrrolidone (PVP) aqueous solution (30 wt%) at a ratio of 1 : 2 and injecting the mixed solution onto a Si substrate by the electrospinning method. Fig. 2 shows the results of observation with the SFT. In the LSM observations in (a) to (c), a condition of complex intertwining of the fibers can be observed. When the area shown by the yellow square at the center of (c) is observed with the SPM, the fine shape of the fibers can be seen from the height image (d). In the phase image (e) of the same view as (d), the difference in the physical properties of the CNF and PVP can be seen. They are represented by the different colors of the yellow region and brown region (e.g., part indicated by the red arrow). Looking at the overlaid image (f) of images (d) and (e), the fact that the outermost surfaces of the fibers are not uniform can be understood from the distribution of the yellow and brown regions on the fibers.

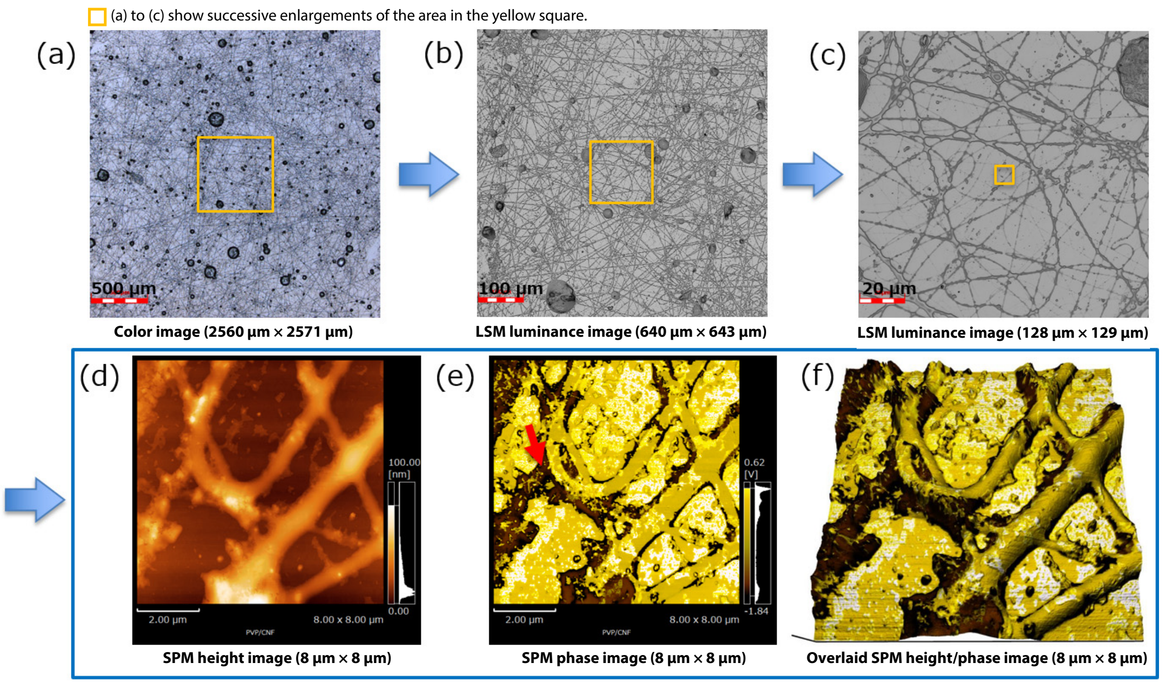


Fig. 2 SFT Images of CNF/PVP Composite

3D Mapping of Young’s Modulus of Composite and Single Materials

3D mapping of the Young’s modulus of the CNF/PVP composite material and the PVP single material was performed with the SPM. Fig. 3 shows images of the Young’s modulus images overlaid on the 3D topographic images. The number of data points of both (a) CNF/PVP composite and (b) PVP single material is 128 × 128. The numbers attached to the images correspond to the numbers of the measurement points in the table at the right.

The positional relationship between the fibers and the distribution of Young’s modulus can be seen clearly in (a). There are regions showing Young’s modulus of approximately 100 MPa (light blue) and regions showing approximately 300 MPa (yellow). From (b), the Young's modulus of the single PVP is around 100 MPa. Therefore, the regions shown by 1 to 5 in (a) are considered to be areas where PVP exists on the outermost surface, while the regions shown by 6 to 10 are areas where CNF exists on the outermost surface.

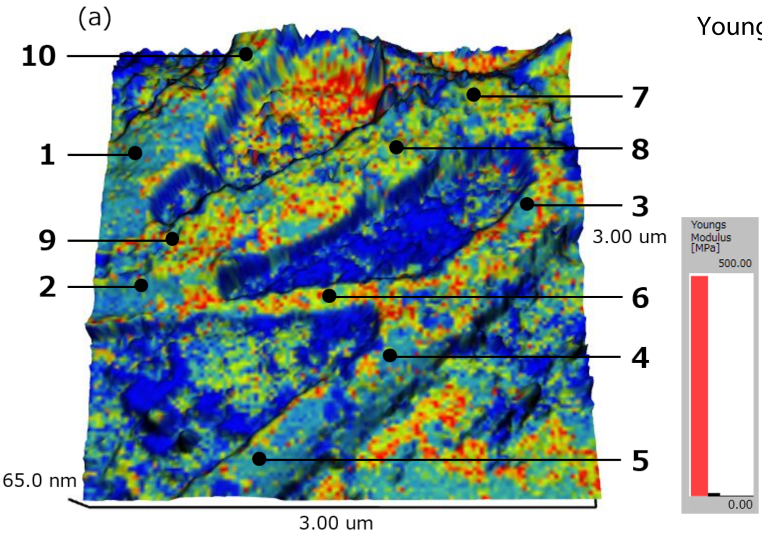
As demonstrated here, visualization of the dispersion state of CNF and PVP was possible by using Nano 3D Mapping.

Conclusion

A wide range of information was obtained by SFT observation, from the overall shape of the fibers to their microtopography, and the distribution originating from differences in the physical properties of the CNF and PVP was also visualized. It was possible to characterize the dispersion states of CNF and PVP by mapping of the Young's modulus by SPM.

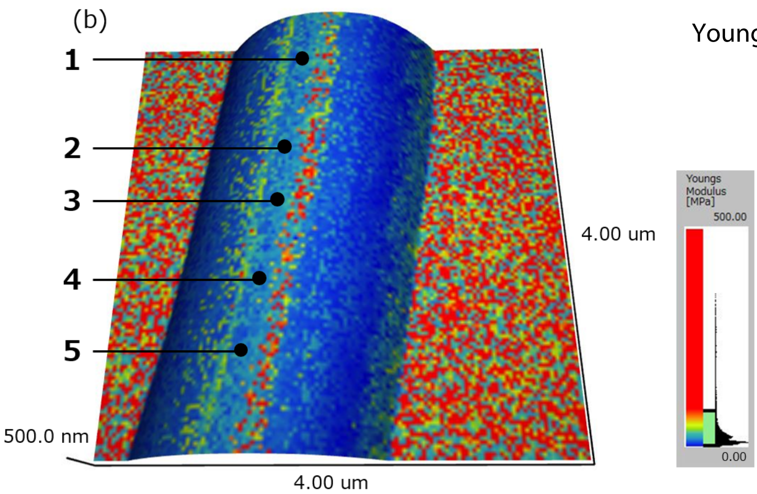
In the future, application of this technique to various composite materials containing CNF and polymers is expected.

<Acknowledgments>
The sample materials used here were provided by Prof. Takahisa Nakai of Mie University and Researcher Keisuke Toba of the Forest Research and Management Organization.



Young’s Modulus at Measurement Points

Measurement Point	Young’s Modulus (MPa)
1	107
2	102
3	121
4	104
5	120
6	274
7	324
8	256
9	319
10	287



Young’s Modulus at Measurement Points

Measurement Point	Young’s Modulus (MPa)
1	99
2	125
3	106
4	108
5	117

Fig. 3 3D Mapping of Young’s Modulus
(a) CNF/PVP Composite, (b) PVP Single Material

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