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Application News

No. S53

Scanning Probe Microscope SPM / Atomic Force Microscope AFM

Approaches to Nanoparticles by SPM Observation and Size Distribution Analysis of Nanoparticles with Mixed Sizes

Introduction

Nanoparticles are used in diverse fields, including magnetic data memory, photocatalysts, biosensing, and drug delivery systems (DDS). Because the shape, size, and other features of nanoparticles govern their product performance, strict quality control is necessary. For example, in case nanoparticles are to be used as an abrasive when manufacturing silicon wafers or other semiconductor substrates for microchips, uniform particle size is essential, and in applications such as biosensing which utilize the optical characteristics of nanoparticles, not only the size but also the shape of the nanoparticles is important.

The scanning probe microscope (SPM) is a type of microscope which is capable of observing the 3-dimensional topography and local physical properties of samples by scanning the sample surface with a minute probe (cantilever). Boasting nanometer order resolution, SPM enables detailed observation of the shapes of nanoparticles and measurement of particle diameter. Moreover, because conductivity is not required in samples, it is also possible to analyze nanoparticles of various materials, including silica, metals, polymers, and biomaterials, without selecting the atmosphere for observation and measurement. This article introduces an example of shape observation and analysis of the particle size distribution of nanoparticles by SPM.

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SPM-9700HT™ Scanning Probe Microscope

Fig. 1 shows the appearance of the SPM-9700HT scanning probe microscope system. Here, Shimadzu optional "particle analysis software" was used in the analysis of nanoparticles. The particle analysis software is capable of extracting multiple particles from the image data observed with the SPM-9700HT, calculating feature values such as the height and surface area of individual particles, and analyzing the results.

Fig. 1 SPM-9700HT™ Scanning Probe Microscope System

Observation of Nanoparticles with Different Shapes

Two types of gold nanoparticles with different shapes (spherical, spike-shaped) were observed with the SPM. Fig. 2 shows a 3-dimensional topographic image of a spherical particle, and Fig. 3 shows a topographic image of a spike-shaped particle. The differences in the particle shape are clearly visible. The particle diameter of the spherical particle in Fig. 2 was 100 nm, while that of the spike-shaped particle in Fig. 3 was 120 nm. Thus, the SPM makes it possible to observe nanoparticles with a diameter of only 100 nm with sufficiently high resolution to capture detailed differences in surface topography.

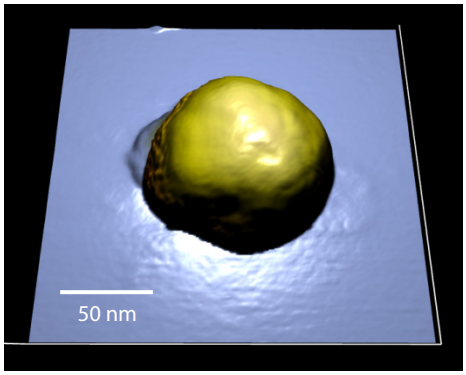


Fig. 2 Topographic Image of Spherical Particle (Range of View: 200 nm × 200 nm, Particle Diameter: 100 nm)

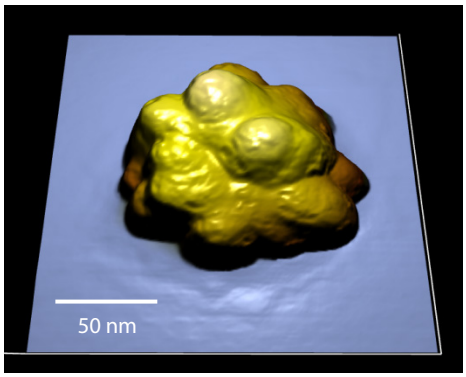


Fig. 3 Topographic Image of Spike-Shaped Particle (Range of View: 200 nm × 200 nm, Particle Diameter: 120 nm)

Size Distribution Analysis of Nanoparticles with Mixed Sizes

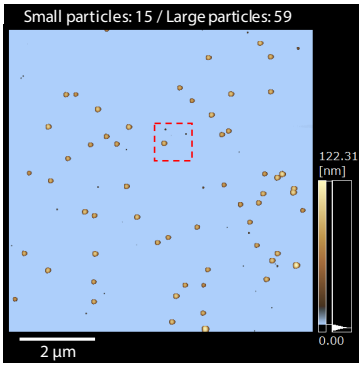
Nanoparticles with a mixture of large and small sizes were observed, and a particle size distribution analysis was conducted. In addition to SPM, various other analytical instruments can also be used to evaluate the sizes of nanoparticles, including the laser diffraction particle size analyzer (SALD™), the induced grating type single nano particle size analyzer (IG), dynamic light scattering (DLS) instruments, the scanning electron microscope (SEM), and the transmission electron microscope (TEM). The most suitable analytical instrument is selected depending on the particle size and material. As mentioned above, because conductivity is not required in SPM samples, analyses of nanoparticles of various materials can be carried out without selecting the atmosphere for observation and measurement. In addition, the greatest advantage of SPM in particle size distribution analysis, even in case nanoparticles with mixed sizes, is the ability to identify clearly the particle diameters of individual particles. In applications that require a uniform particle diameter, it is essential to check whether the material contains particles with mixed sizes different from the target size.

Here, six different locations were observed using the SPM. Fig. 4 (a) shows a typical result of shape observation, and Fig. 4 (b) shows an enlarged view of part of (a). The range of view in (a) is 8 µm × 8 µm, and that in (b) is 1 µm × 1 µm. In (b), individual nanoparticles with different sizes can be clearly observed, and small particles with sizes on the order of 20 nm are also captured without omission.

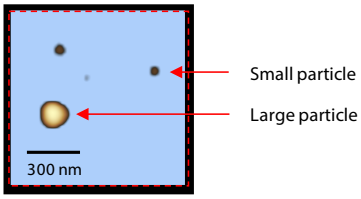
Particles were extracted from the topographic images of the six fields of view with the 8 µm × 8 µm range by applying the particle analysis software. Fig. 5 shows the particle size distribution. To ensure accurate measurement, particles which were not entirely within the range of view were excluded from the analysis. A total of 416 nanoparticles were extracted. The particle analysis software makes it possible to classify multiple extracted particles by feature values. In this analysis, the particle diameter of 60 nm was set as a threshold value, and particles with diameters 60 nm or smaller were classified as small particles, while those with diameters of 60 nm or larger were classified as large particles. As a result of this analysis, the average diameter of the small particles was 18.7 nm (119 particles), and the average diameter of the large particles was 95.6 nm (297 particles).

Conclusion

High resolution shape observation of nanoparticles and an analysis of the particle size distribution of nanoparticles with a mixture of large and small sizes were conducted using an SPM. This technique is not limited to observation and measurement of nanoparticles, but can also be used widely with a diverse range of nanomaterials, for example, in evaluations of the fiber diameter and fiber length of nanofibers.



(a) Topographic image of nanoparticles (range of view: 8 µm × 8 µm)



(b) Enlarged view of area outlined by red broken line (range of view: 1 µm × 1 µm)
Small particles with sizes on the order of 20 nm are also captured without omission.

Fig. 4 Topographic Images of Nanoparticles

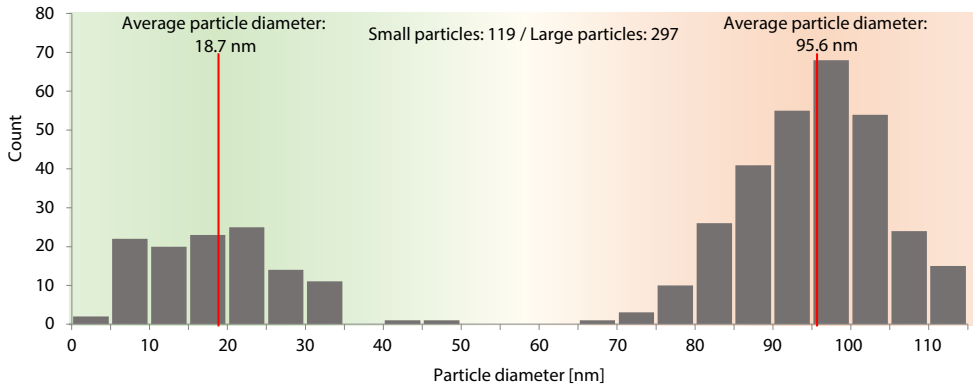


Fig. 5 Particle Size Distribution

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