Establishment of Reasonable coating processing of sample for scanning electronic microscope(SEM) measurement of nano material

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ABSTRACT

The scanning electron microscope has a high resolution, image measurement and micro-zone analysis functions, and is a powerful means in analyzing nanomaterials. However, it is very difficult to measure nanomaterials under tens of nanometers from nanomaterials with deep concave convexes, including nano powders. ㎛-grade porous nanomaterials, the image quality of the scanning electron microscope is improved by the composite coating method of C and Au. [3]
Here, a reasonable sample coating process for improving the image quality in the scanning electron microscopic measurement of the nanomaterial was established.

Keywords

nano material, coating processing of sample, SEM

1. Apparatus, coating materials and test methods

1) Apparatus and coating materials

A Quanta 200 type scanning electron microscope was used as the image measuring device. SCD005 type cathode scattering device and CEA035 type C deposition device were used as sample covering devices. Au, Pt, Ag, and C were used as coating materials.

2) Experimental method

Coating of Au, Pt and Ag materials on the samples was carried out by scattering. The scattering thickness is ensured by adjusting the scattering time, scattering current, distance between the large anode and the sample. [One]

The coating of C on the sample proceeded by the method of deposition. C deposition thickness is guaranteed by adjusting the distance between the carbon fiber and the surface of the sample [2]

The image quality evaluation was carried out by the method of measuring the minimum decomposition distance. The minimum resolution distance measurement was performed by measuring the smallest distance of the lip from the image. The distance measurement was carried out by the "analySIS" program.
The steering constant according to the image measurement of the scanning electron microscope was an acceleration voltage of 30 kV, a working distance (distance between the bottom of the negative electrode and the focal point) of 10 mm, an electron beam diameter of 2.5 nm, and an enlargement magnification of 120,000 times.

Nano ZnO powder prepared by precipitation method was used as a sample.
2. Results and discussion
1) Influence of coating material and thickness on minimum decomposition distance
The effect of the coating material and thickness on the minimum resolution distance of the scanning electron microscopic image was investigated and the results are shown in Fig.

In Figure 1, the curves are the graph of the function when the experimental values are approximated by 4th or 5th order polynomials.

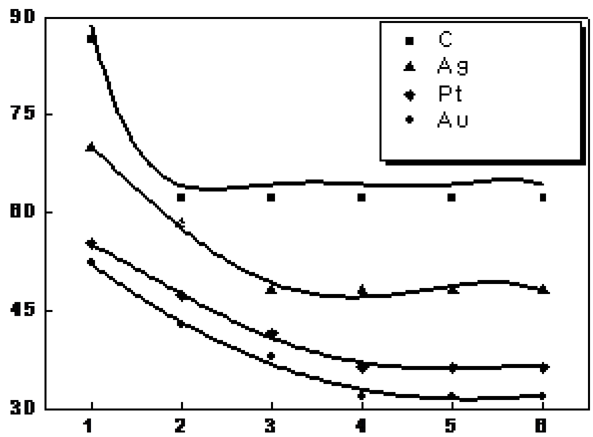

Figure 1. Effect of Coating Material and Coating Thickness on Minimum Disintegration Distance

The figure shows that the minimum decomposition distances in the secondary electron images measured according to the types of coating materials used in the experiment under the same coating thickness conditions are different, showing that Au is the smallest, and Pt, Ag, and C are smaller in order.

Experimental results show that the minimum decomposition distance can be reduced to 62.2 nm for C deposition, and 31.9 nm, 36.4 nm and 48.2 nm for Pt, Au and Ag scattering, respectively.

The reasonable C deposition thickness is 2 nm, and the scattering thicknesses of Au, Pt, and Ag are 4 nm, 4 nm, and 3 nm, respectively.

It can be seen that the minimum decomposition distance is relatively small when Au and Pt are scattered among the coating materials studied in the experiment.

It can be seen from the scanning electron microscopic analysis of the nano powder sample that it is difficult to observe the structure smaller than 30 nm by only the method of scattering Au or Pt when the sample is coated.

2) Influence of sample coating angle on the minimum decomposition distance

In order to investigate the shadow effect depending on the kind of the sample covering material, the minimum decomposition distance of the scanning electron microscopic image according to the sample covering angle was measured and the result is shown in Fig.

C deposition thickness was 2 nm, and the scattering thicknesses of Au, Pt, and Ag were 4 nm, 4 nm, and 3 nm, respectively. The specimen covering angle was the angle of the surface on which the sample was installed and the angle of the drum. When coating, the sample covering band was rotated at a speed of 12 times / min.

In Fig. 2, the curves are graphs of the function approximating the experimental points by the second order polynomial.

Considering the minimum values in the curves of Fig. 2, y = 31.0413nm when θ = 67.5 ° and θ = 60.9 ° when Pt scattering, y = 46.2999 when Ag = 67.299 when Ag scattering nm, and C = 54.495nm when C = 56.4 ° for C deposition.

The curves in Fig. 2 show that the dependence of the minimum decomposition distance on the sample coating angle differs depending on the type of coating material. It can be seen that the minimum decomposition distance of the obtained secondary electron image is hardly affected by the coating angle.

When Ag, Pt, and Au are scattered, the sample coating angles are 65 to 70 °, 60 to 65 ° and 55 to 60 °, respectively.

3) Establish a reasonable sample coating process

C composite deposition method combining Au deposition or Au deposition or Pt deposition is applied to nanomaterials with deep convex convexities on the surface. First, a C-Au or C-Pt composite coating was formed by depositing C at a thickness of 2 nm on the sample and scattering Au or Pt at a thickness of 1, 2, 3, 4, 5 and 6 nm. Coating of Au and Pt was done by scattering method and the angle of scattering was 60 °. The minimum decomposition distance of the scanning electron microscopic image according to the coating thickness of Au and Pt was considered and the results are shown in Fig. In the figure, the dots represent experimental values, and the curves are graphs of approximate functions for experimental values.

In the case of a C-Au composite coating, the coating thicknesses of C and Au are 2 nm and 2.7 nm, respectively, and coating angles are 90 ° and 55 to 60 °, respectively. In the case of the C-Pt composite coating, the coating thicknesses of C and Pt are 2 nm and 2.4 nm, respectively, and coating angles are 90 ° and 60 to 65 °, respectively.

Nano SiO_2 powder, nano-CaO powder, and nano-TiO_2 powder were covered with the sample coating process established by us and compared with the minimum decomposition distance when covering the result of the measurement of the minimum decomposition distance by the conventional process.)

The established sample coating process was carried out by the composite coating process of C-Au system. In comparison with the previous sample coating process, the scattering thickness of Au was 5 nm and the scattering angle was 90 °.

Table 1. Minimum Disintegration Distance of Several Nano Powders

Sample type division	Nano SiO_2 powder	Nano CaO powder	Nano TiO_2 powder
Conventional sample coating process	**34.6nm**	**32.6nm**	31.8 nm
Established sample coating process	**14.7 nm**	**14.5 nm**	14.5 nm
Before and after establishing the process The ratio of the mini mum decomposition distance	2.35	2.25	2.19

As shown in the table, a reasonable sample coating process for improving the image quality of the scanning electron microscope was established in the nanomaterial, and the minimum decomposition distance for the nano-SiO2 powder, nano-CaO powder, and nano-TiO2 powder was changed from 31.8 to 34.6 nm to 14.5 to 14.7 nm Respectively. This results in the quality of the image being two times higher than that of the past.

A conclusion

We have established a reasonable sample coating process for improving the image quality in scanning electron microscope measurement of nanomaterials.

For several nano powders, the quality of the image was improved by more than two times.

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