Research Article

Study of the Effect of the Image Scanning Speed and the Type of Conductive Coating on the Quality of Sem-Micrographs of Oxide Nano Materials for Medical Use

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Abstract

Within the framework of this work, the results of the study of ZnO and SiO_2 samples by scanning electron microscopy at various scanning parameters are presented. It is established that the quality of the resulting image is affected by the speed of image scanning and the presence of an additional conductive coating. When studying ZnO nanoparticles, it is shown that with an increase in the image scanning speed from 1 μs/pixel-7 μs/pixel, the number of aberrations decreases, which leads to a higher contrast of the ZnO particle boundaries. In the case of SiO_2, with an increase in the scanning speed of the image, a decrease in the contrast of the boundaries is observed, which is associated with the accumulation of electron beam charges on the sample surface. It is shown that when a layer of gold is deposited on the surface of the sample, the sharpness of the resulting image increases. The deposition of a carbon layer led to the formation of a fuzzy image of the surface of the samples, which is characterized by a fuzzy separation of the particle boundaries of the studied samples.

Keywords: Scanning electron microscopy; Aberration; Conductive coating; Scanning speed; Zinc oxide; Silicon oxide

Introduction

Scanning Electron Microscopy (SEM) is an analysis method that allows obtaining enlarged images of objects up to subnanometer sizes. Images of the objects under study in this method are formed as a result of scanning the sample with a focused beam of primary electrons. The scanning electron microscopy method is widely used in various fields of scientific and practical activity, such as metal science, chemistry, medicine. [1-9] Due to its high information content, simplicity of sample preparation, as well as automated quantitative analysis and processing of measurement results, the scanning electron microscope is a universal device for studying the structure and topography of the surface of materials. [10-19]

The dispersion of powders is successfully studied on SEM, unlike other types of microscopes; this method allows us to observe the highly porous structure of powders at different stages of production. SEM is used for conducting fractographic studies and for obtaining cannulation patterns. The effect of compositional contrast allows you to observe and identify different phases of the sample.

The scanning electron microscope also provides for the installation of special prefixes for obtaining additional information about the materials under study. [20-27] For example, X-ray radiation is used to assess the chemical composition of a material, cathode luminescence allows you to determine the inclusions and phase composition of semiconductor and non-metallic materials. The SEM method also allows you to register magnetic fields and identify the domain structure of materials. The large depth of the SEM focus allows us to study the kinetics of processes in the sample under external influences (mechanical loads, magnetic and electric fields, chemical reagents, temperature). Currently, up to 60 consoles of various functional purposes can be used for SEM.

Materials and Methods

Samples of silicon dioxide and nanoscale zinc oxide were obtained, respectively, by the Stober method and sol-gel technology.

A more detailed description of the method for obtaining silicon

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dioxide by the Stober method is presented in the following works. [28,29] For the synthesis of SiO_2 microspheres, deionized water was used, the conductivity of which was 0.12 µs/0.2 µs, as well as absolute ethyl alcohol (99.9% volume), and the boiling point was 78.39°C.

The synthesis of nanoscale zinc oxide consisted of the following main stages:

1. Dissolution of a zinc-containing precursor in distilled water.

2. Synthesis of zinc oxide sol.

3. Transfer of sol to gel.

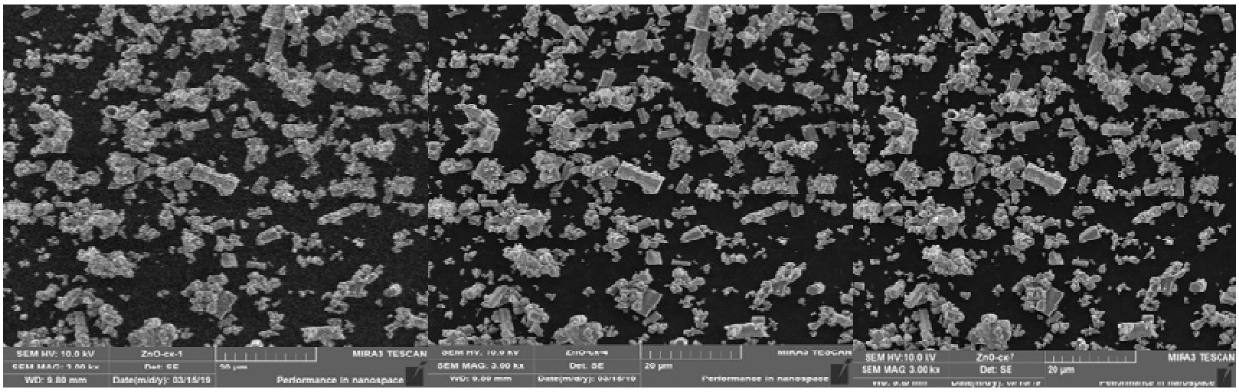
4. Centrifugation of the resulting gel and its washing with distilled water.

5. Drying the gel at a certain temperature.

A detailed method for the synthesis of zinc oxide by the sol-gel method is presented by Blinov et al. [30] Further, the obtained samples of silicon dioxide and nanoscale zinc oxide were examined on a scanning electron microscope "MIRA-LMH" by Tescan. The method of studying samples on a scanning electron microscope was as follows. A double-sided conductive carbon tape was applied to a standard instrument table (12 mm). A thoroughly mixed sample was taken from a dry powder ground in a mortar and applied to the entire area of the adhesive tape. Then carbon was sprayed with a layer of about 20 nm on the QR 150 sputtering system, and gas (nitrogen) was supplied to the microscope system. Next, nitrogen was injected into the working chamber of the microscope; a standard table with a sample was installed in the sample holder. The system was evacuated. The valve of the gun was opening. The selection of parameters for studying the morphology of particles was carried out according to the task. A classical secondary electron detector SE was used to study the parameters affecting the quality of microphotographs

Results and Discussion

When studying the factors affecting the scanning quality, when changing the scanning speed of SEM microphotographs, it is necessary to take into account that the image quality depends on the signal-to-noise ratio of the image. And the signal-to-noise ratio, in turn, depends on how many electrons will be collected to create an image. A very small beam with a low current can have a very good nominal resolution, but noise will be superimposed on the image. Usually, when the TV is scanned to full screen, the image has very poor quality, which makes it difficult to find

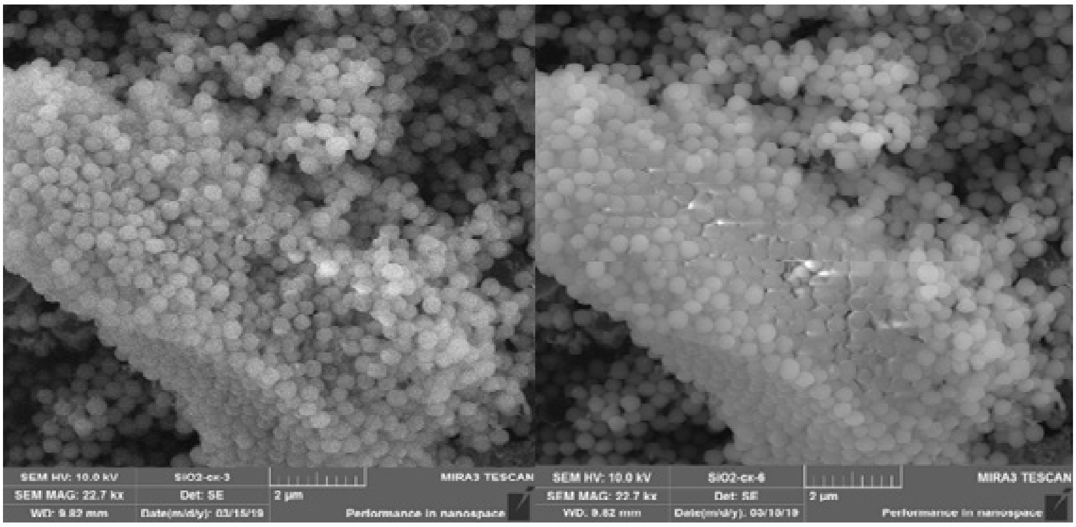


a) 1 = 0.1 µs/pixel

b) 4 = 3.2 µs/pixel

c) 7 = 100 µs/pixel

Figure 1: SEM-micrographs of the ZnO sample using the SE detector at a magnification of 3 thousand times with different scanning speeds.



a) 3 = 1 µs/pixel

b) 6 = 32 µs/pixel

Figure 2: SEM micrographs of a SiO_2 sample using an SE detector at a magnification of 22.7 thousand times at different speeds.

objects. The signal-to-noise ratio can be increased by reducing the scan area (*i.e.* using a small raster), or by increasing the scan time (*i.e.* using a slow scan). A small raster is usually used for focusing, adjusting and correcting astigmatism, while a slow scan is usually used to get a good image. The time of a slow scan frame can take from several seconds to minutes, but this time can be limited by the drift of the object table or the accumulation of charge on the sample surface.

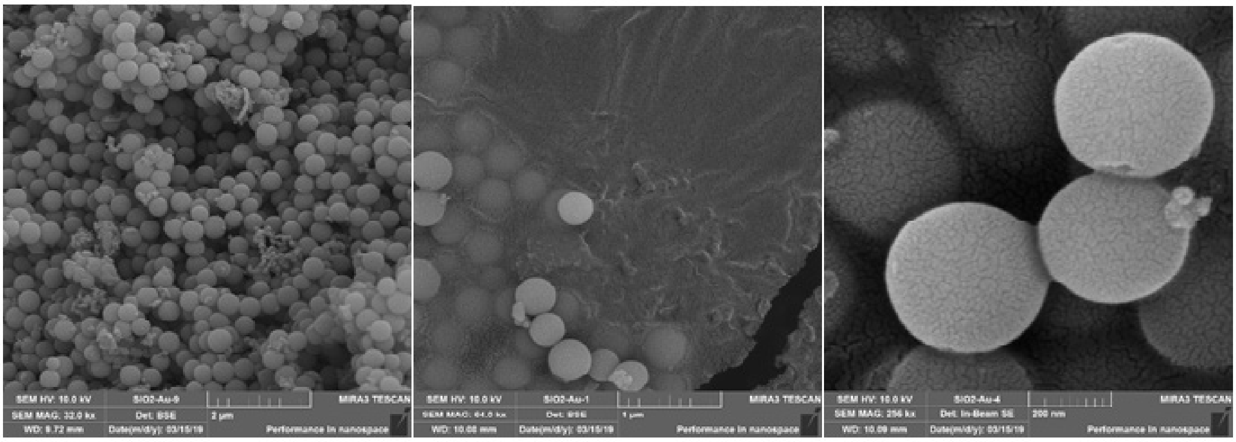
Figure 1 shows micrographs of the ZnO sample using the SE detector at a magnification of 3 thousand times at different speeds. The analysis of microphotographs of the ZnO sample [Figure 1] showed that with an increase in the image scanning speed from 1 to 7 (that is, with a decrease in the scanning time), the number of aberrations (image error in the optical system caused by the deviation of the beam from the direction in which it should go in an ideal optical system) that affect the quality of the photo, which leads to a higher contrast of the boundaries of ZnO particles. But we would also like to take into account that when the image scanning speed increases, a charge from the electron beam can accumulate on the surface of the samples, which leads to”illumination” of the sample surface.

Figure 2 shows micrographs of a silicon dioxide sample using an SE detector at a magnification of 22.7 thousand times at different speeds.

Figure 2 shows micrographs in which, with an increase in the image scanning speed, the accumulated charge of the electron beam on the surface of the SiO_2 microspheres lowered the contrast of the boundaries, which led to a decrease in the quality of the photo. Another way to improve the quality of the obtained microphotographs of the surface of the studied samples is the use of conductive coatings.

Due to the fact that the samples under study may consist of non-conducting materials, a charge may accumulate on their surface as a result of the continuous action of an electron beam. This phenomenon significantly worsens the quality of scanning: highlights of surface areas are formed, the image can “float”, and focusing on the elements of the studied samples is complicated.

To improve the image quality, when scanning SEM, conductive coatings can be applied to the test samples, ensuring the removal of electric charge from their surface. An alloy of gold with palladium is often used, since this material has a small grain size. Chromium, platinum, iridium and carbon are also used. [31-33] Recently, iridium coating has become popular, since it gives a very smooth surface. [31] Carbon, in general, is less effective than other materials, but its advantages are that it does not interfere

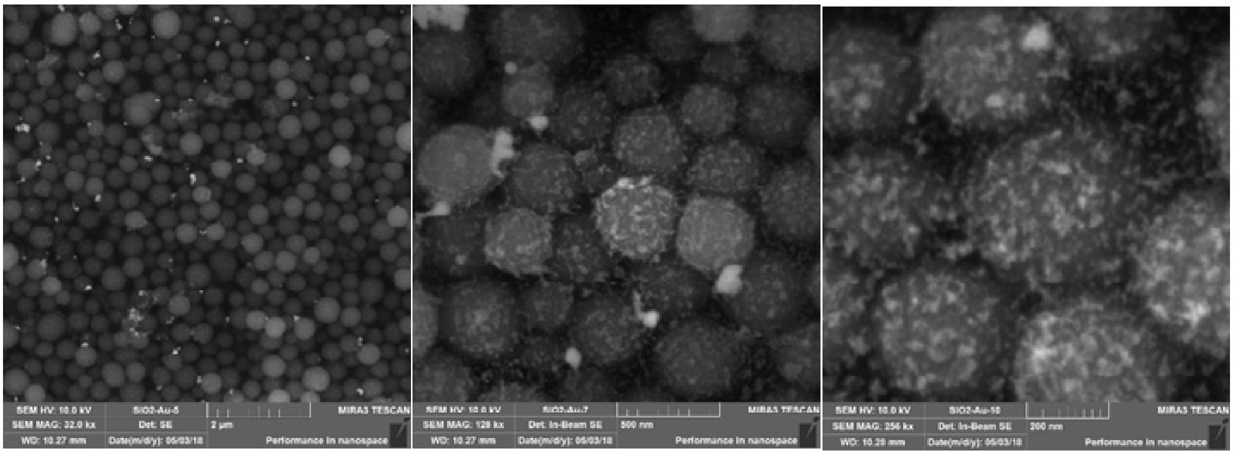


a) An increase of 32 thousand times

b) 64 thousand times

c) 256 thousand times

Figure 3: SEM-micrographs of a SiO_2 sample with a coating of a conductive material (Au).



a) 32 thousand times

b) 128 thousand times

C) 256 thousand times

Figure 4: SEM-micrographs of a SiO_2 sample with carbon deposition in increase.

with X-ray microanalysis and can be easily removed in oxygen plasma. For a solid metal coating, a layer with a thickness of about 10 nm is required, but already 2 nm-3 nm coatings can lead to significant changes in the surface of the sample.

In fact, a conductive coating has three advantages that improve the image. First, it prevents the accumulation of charge on the surface of the sample. The second increases the emission of secondary electrons, which increases the signal and improves the signal-to-noise ratio in the image. Third, it reduces the spread of secondary electrons, which increases the topographic contrast. The latter two effects often improve the image on both conductive and non-conductive samples, even if a very thin (<5 nm) coating is used. Thus, a conductive coating applied to the sample is often used to improve the resulting image. However, it should be borne in mind that the applied conductive coatings can hide the surface relief of the sample under study and make it difficult to find small elements of the system under study, which can be critical when performing some tasks. [34,35]

Figure 3 shows micrographs of silicon oxide microspheres with a conductive material (Au) deposition. The analysis of Figure 3 showed that when a conductive material, such as gold, is sprayed, the sharpness of the resulting image increases. But when the resolution of the microscope is increased, the fine relief of the sample structure is hidden.

Studies with carbon deposition on samples [Figure 4] that have good conductivity, which leads to an indistinct separation of the boundaries of the nanoparticles of the studied samples, makes it difficult to determine the relief of the structure of the sample itself, and it is also impossible to say unequivocally about the presence of the second phase.

Conclusion

Thus, it can be concluded that the image scanning speed and the presence of an additional conductive coating significantly affect the quality of the images obtained by scanning electron microscopy. When studying a sample of ZnO nanoparticles, it is shown that with an increase in the image scanning speed from 1 µs/pixel to 7 µs/pixel, the number of aberrations affecting the quality of the photo decreases, which leads to a higher contrast of the boundaries of the ZnO particles. In the case of SiO_2, with an increase in the scanning speed of the image, a decrease in the contrast of the boundaries is observed, which is associated with the accumulation of electron beam charges on the sample surface. It is shown that when a layer of gold is deposited on the surface of the sample, the sharpness of the resulting image increases. The deposition of a carbon layer led to the formation of a fuzzy image of the surface of the samples, which is characterized by a fuzzy separation of the particle boundaries of the studied samples.

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