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FE-SEM Characterization of Some Nanomaterial

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1. Introduction

In 1931 Max Knoll and Ernst Ruska at the university of Berlin built the first electron microscope that use accelerated electrons as a source instead of light source. However, the first scanning electron microscope (SEM) was built in 1938 due to the difficulties of scanning the electrons through the sample. Electron microscope is working exactly the same as the optical microscope expects it use a focused accelerated electron beam [1].

Since the invention of the electron microscope, it became one of the most useful instruments that has an impact in understanding scientific phenomena in different fields, such as physics, nanotechnology, medicine, chemistry biology..etc. Electron microscope has the ability to resolve objects ranging from part of nano-metre to micro-metre compared to light microscope that has a magnification in the range of 1000 and resolution of 200 nm.

In the first part of the chapter, we will describe some of the basics of electronic microscope and its applications. The second part will be dedicated to the results obtained mainly by SEM.

2. Electron microscopy

2.1 Fundamental principles of electron microscopy

The principal of electron microscope is the same as a light microscope but instead of using visible light it use very energetic electrons as a source. However, the resolution of the optical microscope is limited by its wavelength compared to accelerated electrons which have very short wavelength. This is what makes it possible to see very small features.

In electron microscopes, electrons have very small wavelength λ . This wavelength can be changed according to the applied high voltage. Hence, according to Rayleigh's criterion the wavelength λ of an electron is related to the momentum $p=mv$ of the electron by: [2]

$$\lambda = \frac{h}{p} = \frac{h}{mv} \quad (1)$$

where $h = 6 \times 10^{-34}$ J s is the Planck constant, m and v are the mass and velocity of the electron respectively. Since the electron can reach nearly the velocity of light c then we can use the relativistic equations. In this case the electron mass is changing according to:

$$m = \frac{m_e}{\sqrt{1 - (v/c)^2}} \quad (2)$$

where m_e is the rest mass of the electron. The energy eV transmitted to an electron is giving by:

$$eV = (m - m_0)c^2 \quad (3)$$

By using equations 1,2 and 3 the electron wavelength can be written as function of accelerated voltage: [3]

$$\lambda = \sqrt{\frac{1.5}{V(1 + V * 10^{-6})}} nm \quad (4)$$

for example an accelerated voltage of 10 kV will yield a wavelength of 0.0122 nm. The extremely small wavelengths make it possible to see atomic structures using accelerated electrons.

2.2 Interaction of accelerated electrons with the specimen

The electron beam interacts with the specimen reveal useful information about the sample including: its surface features, size and shape of the features, composition and crystalline structure. The interaction of the electron beam with the specimen can be in different ways:

2.2.1 Secondary electrons

If the incident electrons come close enough to the atom then these electrons will give some of their energy to the specimen electrons mainly in the K-shell. As a result, these electrons will change their path and will ionize the electrons in the specimen atoms. These ionized electrons that escape the atoms are called secondary electrons. These electrons will move to the surface of the specimen and undergoing to elastic and inelastic collision until reaching the surface. However, due to their low energy $\sim 5eV$ only those electrons that are close to the surface ($\sim 10 nm$) will escape the surface and then can be detected and can be used for imaging the topography of the specimen.

2.2.2 Backscattered electrons

When the incident electrons hit an atom directly, then they will be reflected or back-scattered. Different atomic type of atoms will result in a different rate of backscattered electrons and hence the contrast of the image will vary as the atomic number of the specimen change, usually atoms with higher atomic number will appear brighter than those have lower atomic number.

2.2.3 Transmitted electrons

If the incident electrons pass through the specimen without any interaction with their atoms, then these electrons called transmitted electrons, these electrons are used to get an image of

thin specimen. Another scattering mechanism called elastic scattered where electrons don't lose their energy these scattered electrons can be used to get information about orientation and arrangement of atoms.

2.2.4 Other interactions

When the atoms bombarded with incident electrons, electrons will be released from these atoms and this will leave the atom in the excited state. In order for the atom to return to the ground state, it needs to release the excess energy. Auger electrons, X-Rays, and cathodoluminescence are three ways of relaxation. The x-ray is used to identify the elements and their concentrations in the specimen by using a technique called Energy-dispersive X-ray analysis (EDX) technique. Chemical analysis can be done by using Auger electrons.

2.3 Types of electron microscopes

There are two types of electron microscopes. Scanning Electron Microscopes (SEM), and Transmission Electron Microscope (TEM), these types of microscopes detect electrons that are emitted from the surface of the sample. The accelerated voltage is ranging from 10kV to 40kV for the SEM. The thickness of the specimen in this case is not important. In addition, the samples to be tested have to be electrically conductive; otherwise they would be overcharged with electrons. However, they can be coated with a conductive layer of metal or carbon.

In TEM the transmitted electrons are detected, and in this case the specimen thickness is important and typically should not exceed 150 nm. The accelerated voltage in this case $\geq 100\text{kV}$.

Since the electrons are easily scattered in air all electron microscopes should operate under a high vacuum.

All types of electron microscopes are basically consist of three basic components:

Electron Gun which is used to provide and supply electrons with the required energy. There are different types of electron gun; the old type was a bent piece of Tungsten wire with 100 micrometres in diameter. Higher performance electron emitters consist of either single crystals of lanthanum hexaboride (LaB₆) or from field emission guns.

3. Experimental

3.1 Pulse Laser Deposition (PLD)

As a materials processing technique, laser ablation was utilized for the first time in the 1960's, after the first commercial ruby laser was invented [4]. Nevertheless, as a thin film growth method it did not attract much research interest until the late 1980's [5], when it has been used for growing high temperature superconductor films. Since then, the development of the pulsed laser deposition (PLD) technique has been more rapid and the amount of research devoted to this topic has increased dramatically [6]. The growth and quality of the resulting film will generally depend on a number of fundamental parameters, including the choice of substrate, the substrate temperature and the absolute and relative kinetic energies and/or arrival rates of the various constituents within the plume.

The PLD process is shown in figure 1:

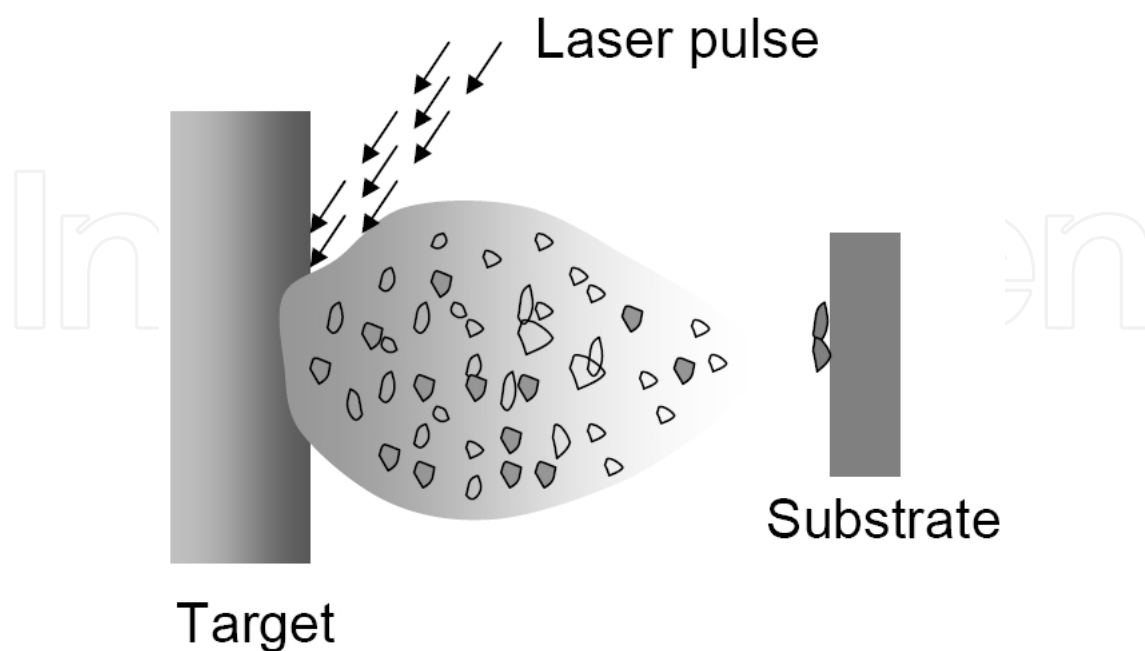


Fig. 1. Schematic presentation of the pulsed laser deposition process

a) Laser – target interaction, b) Plume expansion and c) Film deposition [6].

The growth and quality of the resulting thin film will generally depend on a number of fundamental parameters, including the choice of substrate, the substrate temperature, T_s , distance between target-substrate, pressure and laser energy.

In our case the laser energy was 300 mJ and the time was fixed at 60 minutes. For the others parameters (substrate, substrate temperature, pressure), different values were used.

3.2 Mechanical Alloying (MA)

The ball milling constitutes new promising methods to produce nanosized particles [7,8]. It has many advantages, e.g low cost, simple operation. The ball-milling is generally used as a mechanical co-grinding of powders, Initially different in nature, up to the preparation of a new powder, homogeneous in composition. The milling is done in cylindrical containers called vials and containing balls. The nature of the milling tools can be as diverse as steel, agate, tungsten carbide... The vials are generally filled under an inert atmosphere to avoid side reactions, since the particles are fractured during the milling process and, therefore, new highly reactive surfaces can react with the surrounding gases [8].

Several terms are used to call this technique: "Mechanical Alloying" when there is a chemical reaction between different powders, "Mechanical Grinding" or "Mechanical Milling" when the only goal is to modify the texture and/or the structure of a material (no chemical reaction is involved in the process).

Two kinds of milling systems were used to prepare our nanopowders (Vibrant and planetary milling) and different milling parameters were considered (milling times, balls to powders mass ratio, size of balls and rotation speed).

3.3 Field Emission Scanning Electron Microscopy (FESEM)

The field emission scanning electron microscope (FE-SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. Electron emitters from field emission gun was used. These types of electron emitters can produce up to 1000x the emission of a tungsten filament. However, they required much higher vacuum conditions. After the electrons beam exit the electron gun, they then confined and focused into a thin focused, monochromatic beam using metal apertures and magnetic lenses. Finally, Detectors of each type of electrons are placed in the microscopes that collect signals to produce an image of the specimen.

Particles morphology of our samples was investigated using Nova 200 NanoLab field emission scanning electron microscope (FE-SEM).

4. Results

4.1 Thin film prepared by Pulse Laser Deposition (PLD)

Fig. 2 shows FESEM micrographs of ZnO thin films grown on sapphire substrate by pulse laser deposition at growth temperature from 685 to 750 °C by using a ZnO powder target at high grade. The experimental parameters are summarized in table.1. It is seen that with the substrate temperature increasing the morphology of ZnO thin films have a little difference. The thickness of films decreases with the increase of substrate temperature.

The effect of the distance between target and substrate on the morphology was also studied. Fig. 3 shows the FESEM images of ZnO thin film with different distance between the target and thin film. It is clear that the distance affect the morphology of the film.

TEMPERATURE(°C)	THICKNESS (nm)	Distance between target and the film	Oxygen pressure	LASER ENERGY(mj)	SUBSTRATE
750	510	37.5 mm	150 mTorr	350	Sapphire
700	1230	37.5 mm	150 mTorr	350	Sapphire
685	1115	37.5 mm	150 mTorr	350	Sapphire
400	-	10 mm	150 mTorr	350	Sapphire
400	-	23mm	150 mTorr	350	Sapphire

Table.1.Growth parameters of ZnO thin films

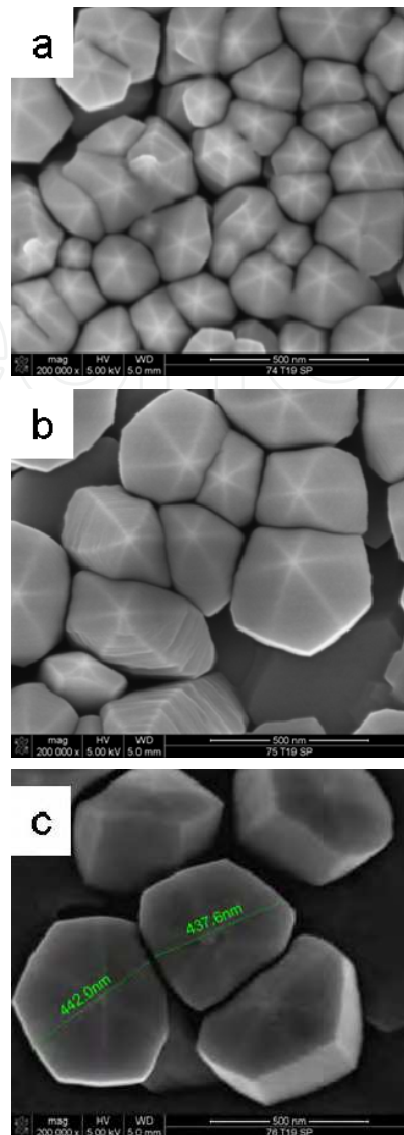


Fig. 2. FESEM images of thin film grown on sapphire at substrate temperature of: (a) 750 °C, (b) 700 °C and (c) 685°C.

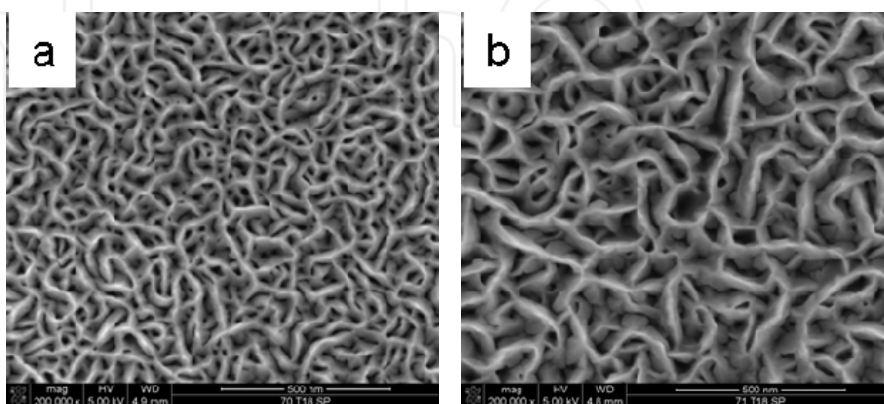


Fig. 3. FESEM images of thin film grown on sapphire at substrate temperature of 400°C: a) distance between target and thin film =10mm and b) distance between target and thin film =23 mm

4.2 Nanopwders obtained by mechanical alloying

4.2.1 Hematite ($\alpha\text{-Fe}_2\text{O}_3$) nanocrystallines

The conditions for production of $\alpha\text{-Fe}_2\text{O}_3$ nano-crystallines by dry milling was studied. [9,10] Commercial $\alpha\text{-Fe}_2\text{O}_3$ powder was used as the starting material. The mechanical milling was carried out in a planetary ball mill Fritsch Pulverisette 6. The powder was ground in vial with 200g of mixture 1:1 in weight of stainless steel balls (10 and 15 mm in diameter). Different milling times were considered (1, 6, 12, 24 and 48h) and the sample to balls weight ratio was fixed to 1:10. The milling intensity was 250 rpm. Fig.4 shows scanning electron micrographs before and after milling. It is clear that un-milled powder shows an in homogeneities regarding particle size distribution (Fig. 4a). After milling, a reduction of the particle size can be observed with relatively better homogeneity (Fig. 4b-d). SEM images for increasing milling times reveal clearly that large particles are in fact agglomerates of much smaller particles.

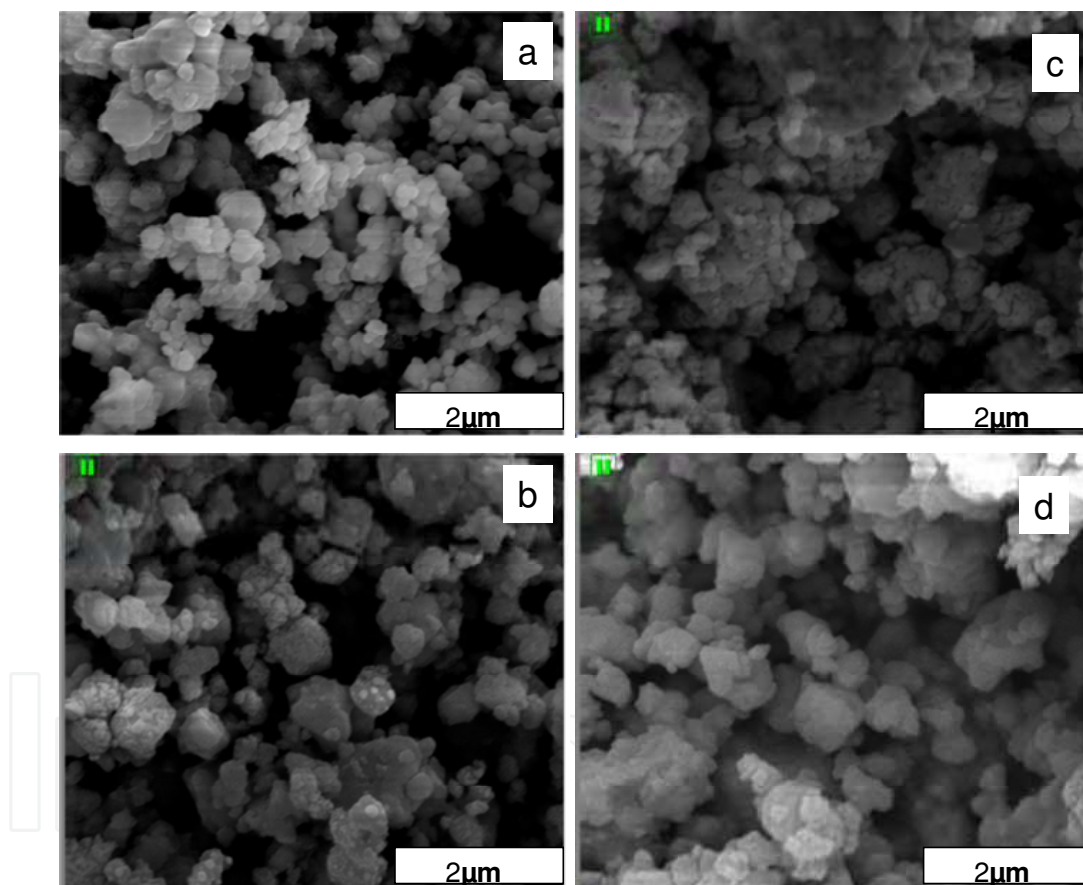


Fig. 4. FESEM images for the samples milled at different times : a) 0h, b) 12h, c) 24h and d) 48h.

4.2.2 Nanocrystalline zinc ferrite (ZnFe_2O_4)

Nanocrystalline zinc ferrite (ZnFe_2O_4) is synthesized by high-energy ball-milling from a powders mixture of zinc oxide (ZnO) and hematite ($\alpha\text{-Fe}_2\text{O}_3$). [11] Commercially powders of hematite ($\alpha\text{-Fe}_2\text{O}_3$) and zinc oxide (ZnO) are used with equal molar (1:1) and were introduced into a stainless steel vials with stainless steel balls (12 mm and 6 mm in diameter) in a high energy mill (SPEX 8000 mixer mill). Different milling times were

considered (6, 12 and 24) and two values of the balls to powders mass ratio were used (10:1 and 20:1). SEM micrographs of the samples before and after milling are shown in Figure 5 It is clear that unmilled powder shows a different shape of powders due to zinc oxides and hematite powders (fig.5a, 5b). After milling, a reduction of the crystallite size can be observed fig.5c. High magnification images (fig.5d) reveal clearly the formation of a new nanocrystalline different from the started materials.

4.2.3 Zinc oxides Nanocrystalline (ZnO)

The effects of milling times on the mechanically milled ZnO powder are also studied [12]. Commercially ZnO powders with average particle size of about 1 μm and 99.9% of purity, were introduced into a stainless steel vials with stainless steel balls (12mm and 6mm in diameter) in a SPEX 8000 mixer mill, then milled for different milling periods of time. The balls to powder mass ratio was fixed to 10:1. SEM micrographs of the samples before after milling are shown in Figure.6. It is clear that un-milled powder shows un-homogeneities regarding particle size distribution, where the average size varies in the range 150 – 800 nm (Fig. 6a, 6b). After milling, a reduction of the particle size can be observed with relatively better homogeneity (Fig. 6c, 6e). High magnification images (Fig. 6d, 6f) reveal clearly that large particles are in fact agglomerates of much smaller particles. The average particle size after milling is less than 100nm.

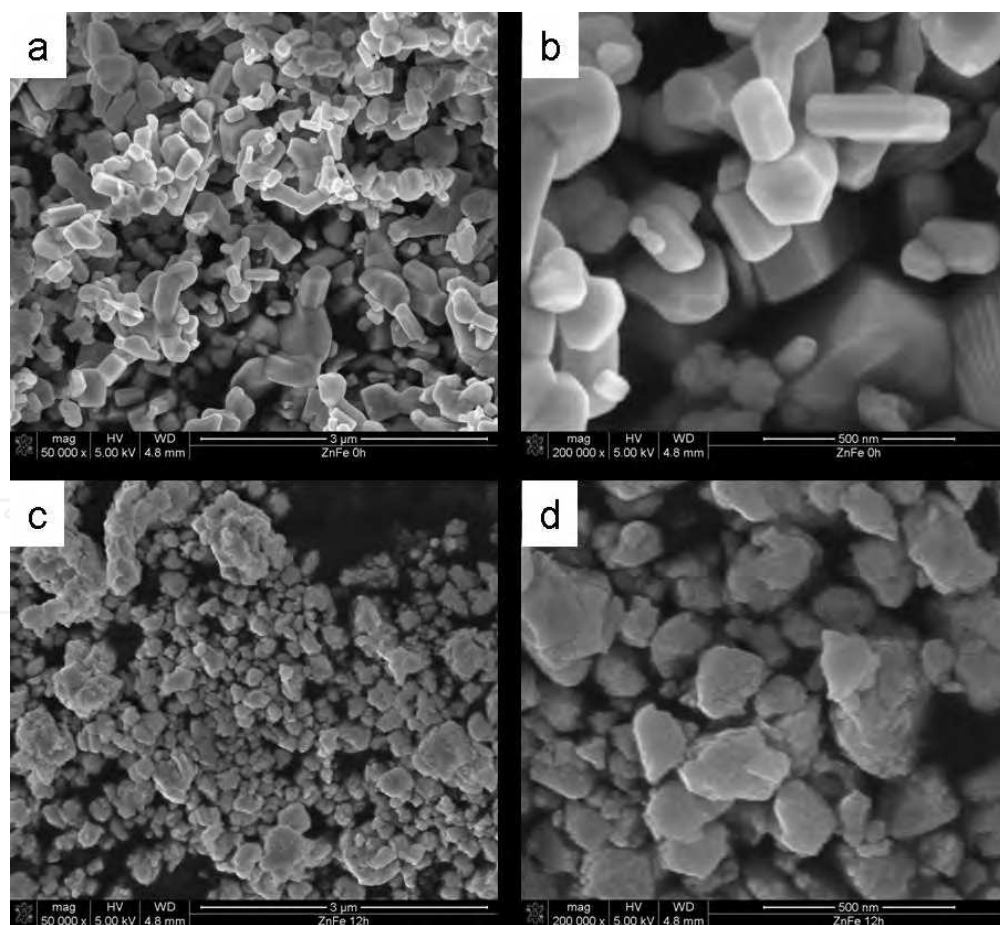


Fig. 5. FESEM micrographs of mixtures (zinc oxides +hematite) powders as received (a) as received at high magnification (b) milled for 12h (c) milled for 12h high magnification (d)

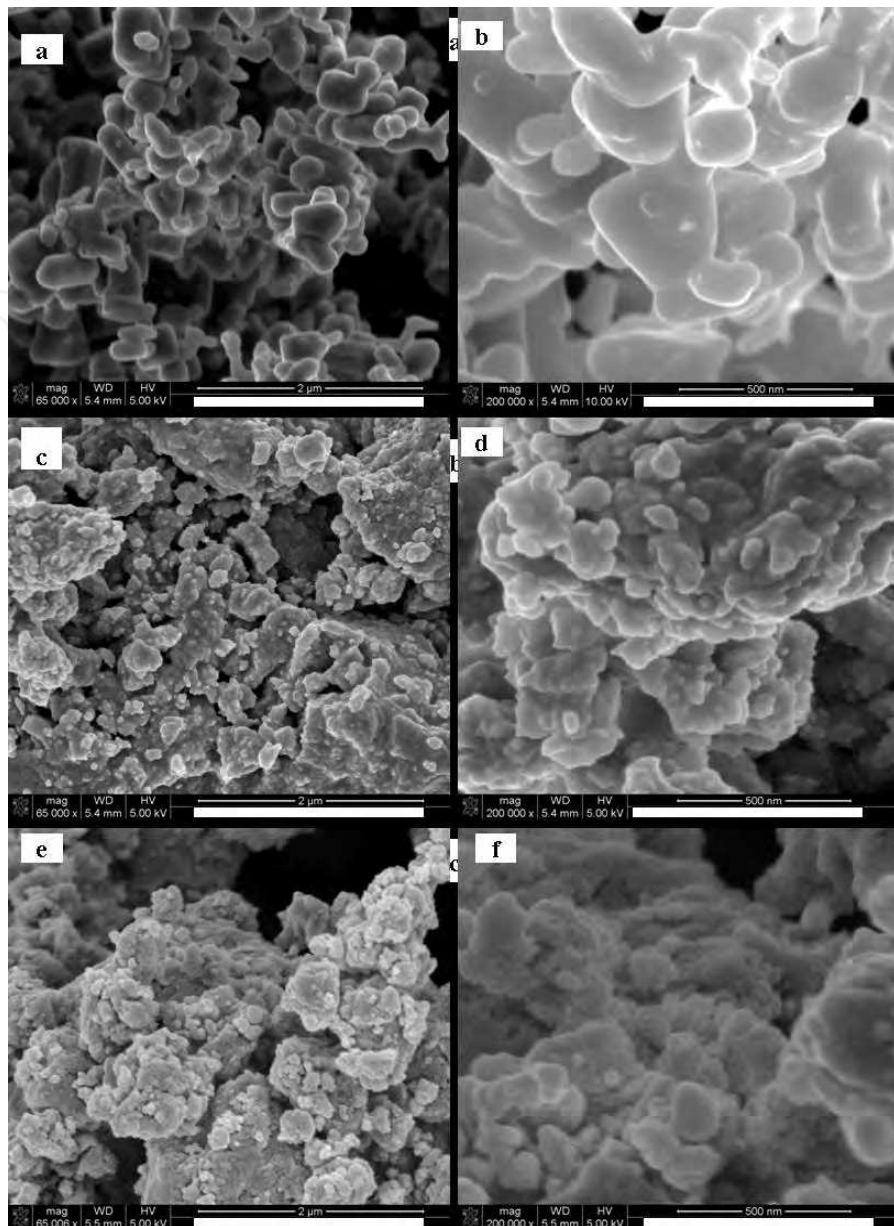


Fig. 6. SEM micrographs of ZnO powder for different milling time: a) as received; b) as received at high magnification; c) milled for 3h; d) milled for 3h high magnification; e) milled for 5h; f) milled for 5h at high magnification.

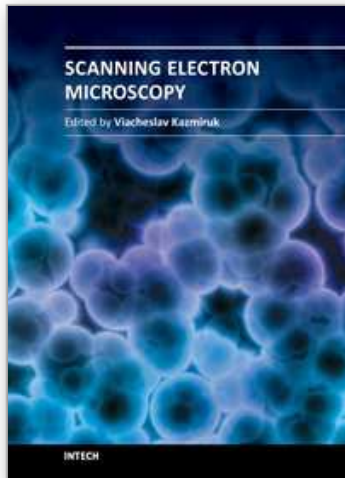
In summary, it is clear that scanning electron microscopy gives tremendous information about the microstructure of nanomaterials including thin film and nano-powders. In addition to that the signals coming from the sample can be used to get information about the composition of the materials and the structure.

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Today, an individual would be hard-pressed to find any science field that does not employ methods and instruments based on the use of fine focused electron and ion beams. Well instrumented and supplemented with advanced methods and techniques, SEMs provide possibilities not only of surface imaging but quantitative measurement of object topologies, local electrophysical characteristics of semiconductor structures and performing elemental analysis. Moreover, a fine focused e-beam is widely used for the creation of micro and nanostructures. The book's approach covers both theoretical and practical issues related to scanning electron microscopy. The book has 41 chapters, divided into six sections: Instrumentation, Methodology, Biology, Medicine, Material Science, Nanostructured Materials for Electronic Industry, Thin Films, Membranes, Ceramic, Geoscience, and Mineralogy. Each chapter, written by different authors, is a complete work which presupposes that readers have some background knowledge on the subject.

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