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ABSTRACT

Zinc Oxide (ZnO) is a crystal that is widely used in various purposes as an additive in paints, ceramic materials, catalysts, electronic equipment, and semiconductors. One application of nanoparticles is the addition of nano zinc oxide (ZnO) to the coating which can increase corrosion resistance higher than without nano ZnO. This research method uses a Planetary Ball Mill with variations of disk rotation speed of 300 and 400 rpm and the use of grinding balls of 5 and 10 mm. From the results of this study, variations in disk rotation speed and the use of grinding ball sizes can affect particle size.

1. INTRODUCTION

Nanotechnology is one of the advancements in the field of materials and engineering that is growing rapidly at this time. The concept of nanotechnology was first started by Richard Feynman in 1959. Nanotechnology is the result of utilizing particle sizes to develop new properties. The results of nanotechnology are called nanomaterials or nanoparticles. The resulting particles have a nanometer size that is 1-100 nm [16]. Metal nanoparticles have attracted the attention of researchers because their application provides many benefits. One application of nanoparticles, namely the addition of nano zinc oxide (ZnO) to the coating can increase corrosion resistance higher than without nano ZnO [20].

Zinc Oxide (ZnO) is a crystal that is widely used in various purposes as an additive in paints, ceramic materials, catalysts, electronic equipment and semiconductors. One of the characteristics of ZnO is that its chemical compounds can combine with other compounds. The manufacture of ZnO nanoparticles can use a planetary ball mill [16]. Planetary ball mill technology is a tool used for the process of crushing materials in a fine level. This research conducted the manufacture of ZnO nanoparticles with the planetary ball mill method. As well as knowing the effect of disk rotation speed and diameter grinding ball parameters on particle characteristics. Then performed using a scanning electron microscope and x-ray diffraction.

2. THEORY BASIS

Nanomaterial or nanoparticle is an element of nanoscience and nanotechnology that has the potential to significantly change materials and produce results so that it can have a very broad impact in technology fields such as electronics, medicine, automotive, and other fields [17]. Nanomaterials are an interesting study, because nanometer-sized materials usually have particles with superior properties compared to larger materials [23]. In general, to form nanoparticles, synthesis can be carried out using a chemical 'bottom-up' method, namely by uniting or growing atoms or by a 'top-down' method, namely making nanomaterials by separating molecules that are gathered in large quantities into small ones by milling techniques. Nanomaterials are very fine particles with a nanometer size. Based on the material of origin, nanomaterials can be classified into two groups, namely organic nanomaterials and inorganic nanomaterials. Organic nanomaterials are carbon nanoparticles, while inorganic nanomaterials include magnetic nanoparticles, precious metal nanoparticles (such as gold and silver) and semiconductor nanoparticles (such as titanium dioxide and zinc oxide).

Zinc oxide or zinc oxide is an inorganic compound with the formula ZnO. This compound is a white powder, almost insoluble in water. ZnO powder is often used as an additive to various materials and products including plastics, ceramics, glass, cement, rubber (such as car tires), and lubricants [11]. Zinc Oxide (ZnO) is a crystal that is widely used in various purposes as an additive in paints, ceramic materials, catalysts, electronic equipment, and semiconductors [11]. ZnO is a semiconductor that has unique properties, namely it has an energy gap of 3.37 eV and an excitation binding energy of 60 meV. One of the characteristics of ZnO is that its chemical compounds can combine with other compounds. The advantages of using ZnO are its low price, abundant supply, stable chemical structure, and non-toxicity. Zinc Oxide crystals have the form of rocksalt, zinc blende, and wurtzite. One method to produce ZnO nanoparticles is by using a planetary ball mill [18].

Ball mills are an efficient and simple method for shaping sub-micron or nano-sized materials. There are many types of milling methods, one of which is the planetary ball mill [21]. A planetary ball mill is a machine tool that plays a very important role because of the characteristics of the ball mill as a material crushing tool with collisions and friction of powder types on a large or small scale. mill that uses centrifugal force to rotate balls and bowls at speeds up to twenty times the acceleration of gravity [19].

A planetary ball mill is made of two or more jars rotating around an axis, mounted on a disk that rotates at a certain speed. Milling occurs as a result of having a jar attached to a disk rotating a common central axis while the jar simultaneously rotates around its own axis. The working principle of the planetary ball mill is that the powder is put into a jar or bowl in which there are several balls and then they rotate continuously. Inside the jar, the balls will collide with each other. As a result of the collision of the balls, the powder that is put into the jar will be crushed or crushed between the balls. This causes the particle to break, and so on until the particle size reaches the desired [1].

The effectiveness of producing the desired powder is determined by several parameters, related to the geometry, physical properties of the tube, and grinding medium, including spherical size and shape, angular velocity, milling time, and load fraction. The homogeneity and contamination of the product from the bowl are also related to the parameters specified above and must be properly accounted for [6]. The milled powder was tested by x-ray diffraction testing. Then observations using a scanning electron microscope to determine the surface of the specimen [24].

3. METHODOLOGY

3.1 Specimen Making

The zinc oxide (ZnO) used is 64 m produced by MERCK. ZnO powder, stearic acid, and grinding balls were put in a bowl and milled using a planetary ball mill with a time of 5 hours and a ball to powder ratio of 20:1 and variations of disk rotation speed parameters of 300 and 400 rpm and the use of grinding balls of 5 and 10 mm. The milled powder is then heated in a furnace at a temperature of 380 C for 30 minutes to remove steric acid. After that it was crushed using a magnetic stirrer then filtered and put into a sample bottle.

3.2 Testing

Scanning Electron Microscope (SEM) observations aim to determine the size of ZnO nanoparticles. To determine the particle size, it is assumed to be spherical, so the area (A) can be calculated to determine (d) the particle diameter [14].

$$d = 2\sqrt{\frac{A}{\pi}} \quad (1)$$

X-ray diffraction testing using the Pananalytical Expert Pro. This test is to determine the diffraction pattern, crystal size, lattice parameters and crystal structure.

To determine the crystal size using the Deybe Scherrer equation [2].

$$D = \frac{K.\lambda}{\beta.cos\theta} \quad (2)$$

D: crystal diameter size (nm)
λ:: X-ray wavelength used, using Cu-Ka 1.540598 (0.1540598 nm)
θ: Bragg angle (o)
β: FWHM (full width at half maximum) one selected peak (radians)
K: material constant whose value is less than one, generally using 0.94

The crystal lattice parameter or the distance between planes in the lattice (d) of a hexagonal system such as ZnO can be calculated using Eq. The lattice parameters a and c were obtained using diffraction peaks with Miller indices (100) and (002) [12].

$$a = \frac{\lambda}{\sqrt{3}\ Sin\theta} \quad (5)$$

$$c = \frac{\lambda}{Sin\theta} \quad (6)$$

4. RESULT AND DISCUSSION

4.1 Scanning Electron Microscope

This test was carried out on the four variations of the sample. Following are the results of the SEM observations of disk rotation speed parameters of 300 rpm with variations in the diameter of the grinding ball 5 and 10 mm with a magnification of 50000x.

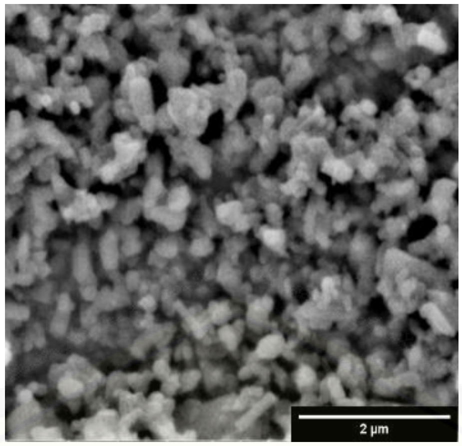


Figure 1. SEM results of 5 mm diameter grinding ball parameters with a disk rotation speed of 300 rpm

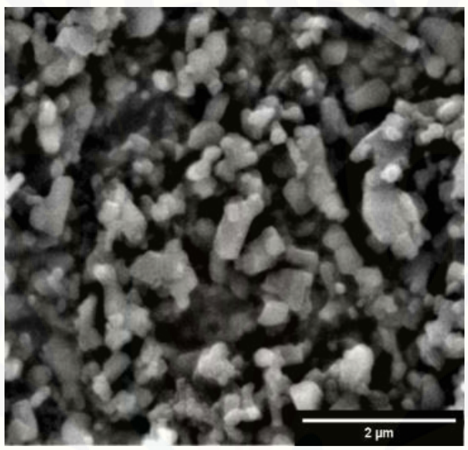
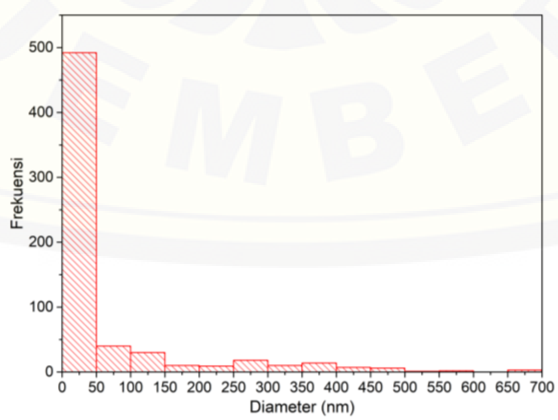
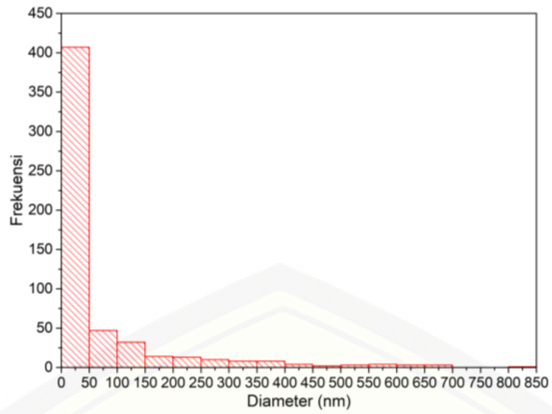


Figure 2. SEM results of 10 mm diameter grinding ball parameters with a disk rotation speed of 300 rpm

The results of the SEM observations in Figure 1 were then analyzed using ImageJ using the analyze particles feature and entered equation 1 for the 5 mm diameter grinding ball parameter with a disk rotation speed of 300 rpm to get an average particle size of 62 nm. While the particle size parameter is a grinding ball diameter of 10 mm with a disk rotation speed of 300 rpm with an average particle size of 71 nm.



(a)



(b)

Figure 3. Particle distribution diagram of parameters (a) 5 mm diameter grinding ball with a disk rotation speed of 300 (b) 10 mm grinding ball with a disk rotation speed of 300 rpm

Figure 2 is a histogram of the particle size distribution. The parameters of the 5 mm diameter grinding ball with a disk rotation speed of 300 rpm and a grinding ball diameter of 10 mm with a disk rotation speed of 300 rpm obtained particles with a size of less than 100 nm reached 82% and particles with a size of more than 100 nm were 18%. The following are the results of SEM observations with a magnification of 50000x parameter disk rotation speed 400 rpm with variations in the diameter of the grinding ball 5 and 10 mm. The image was analyzed using ImageJ software to determine the particle size.

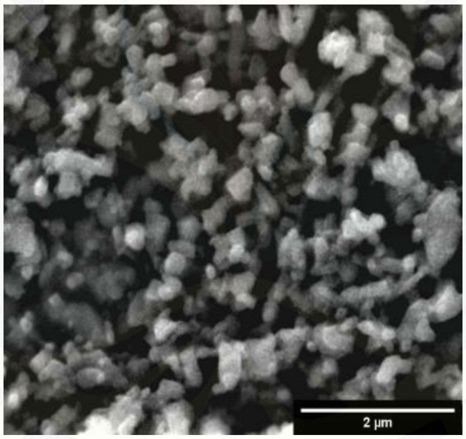


Figure 4. SEM results for the 5 mm diameter grinding ball with a disk rotation speed of 400 rpm

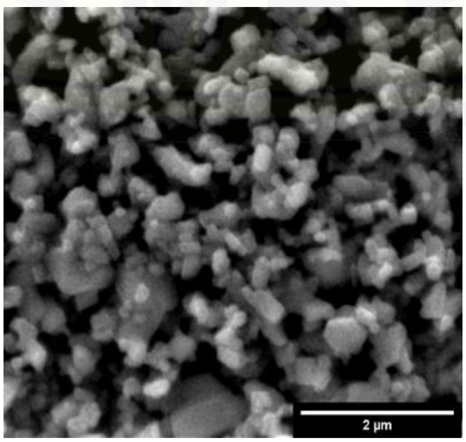


Figure 5. Results of SEM parameters with a 10 mm diameter grinding ball with a disk rotation speed of 300

rpm

The results of the SEM observations in Figure 3 were analyzed by ImageJ using the analyze particles feature and entered equation 1. In the 5 mm diameter grinding ball parameter with a disk rotation speed of 400 rpm, the average particle size is 57 nm. While the diameter of the grinding ball is 10 mm with a disk rotation speed of 400 rpm with an average particle size of 65 nm.

Figure 4 is a particle size distribution diagram. The parameters of the 5 mm diameter grinding ball with a disk rotation speed of 300 rpm and a grinding ball diameter of 10 mm with a disk rotation speed of 300 rpm obtained particles with a size of less than 100 nm reaching 82% and particles measuring more than 100 nm 18%.

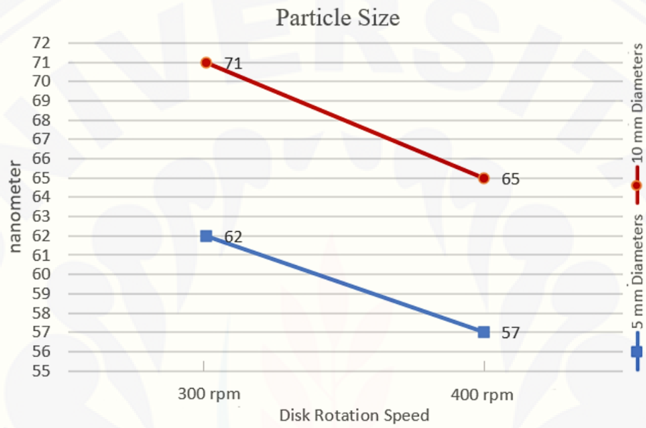


Figure 6. Particle size chart

The results of observations and analysis showed that the particle size of Figure 6 with a diameter of 5 mm grinding ball obtained a smaller size and more homogeneous or uniform than milling with a grinding ball with a diameter of 10 mm. The effect of grinding ball diameter is illustrated in Figure 7.

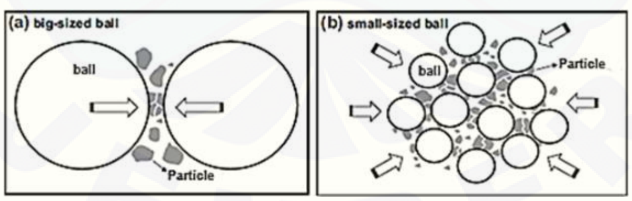


Figure 7. (a) Milling with large balls (b) Milling with small balls [11]

In milling using a grinding ball diameter of 5 mm and 10 mm will have an effect. Different dimensions will of course affect the weight of the grinding ball, so the crushing force of the mill will be different as well. A 5 mm diameter grinding ball with a weight of 0.30 grams has a crushing force of 0.65 N and 1.16 N, while a ball with a diameter of 10 mm weighing 2.35 grams has a crushing force of 4.99 N and 18.86 N for each grinding ball. Due to the difference in diameter and weight between 5 mm and 10 mm grinding balls, it will affect the number of grinding balls in the bowl.

The particles grinded by a ball with a diameter of 10 mm despite having a high crushing force but only part of the particles colliding with the ball due to the large size difference between the ball and the powder particle is illustrated in figure 4.9(a), so that during the collision between the balls grinder with powder only partially exposed so that the grinding of the powder becomes inhomogeneous. Whereas in milling with a ball mill with a diameter of 5 mm with a lower crushing force the particles will be confined in a narrow space surrounded by small balls. This is due to the greater number of balls so that the particles collide evenly with some of the existing balls. It was concluded that with a low collision energy but high frequency it can reduce particles with homogeneous results [13].

In addition to the diameter of the grinding ball, the disk rotation speed also affects the particle size results. Figure 4.9 can be seen that the particle size at a disk rotation speed of 400 rpm is smaller than at a speed of 300 rpm. The crushing force at 300 rpm is 27.60 N while at 400 rpm the crushing force is 49.05 N. In the grinding process, the higher energy in the grinding process causes the collision between the ball and the powder to increase, thus faster speed. can result in a reduction in particle size [4].

In the results of SEM observations, there are particles that occur agglomeration. Agglomeration is the occurrence of clumping or sticking of fine material into a larger size. Agglomeration can occur due to long grinding and high energy during grinding [7]. As a result of long grinding and with very fine particles, the interaction between particles becomes higher, as well as the presence of Van Der Waals forces or attractive forces between molecules so that the particles agglomerate [9].

4.2 X-Ray Diffraction

The XRD diffraction wave pattern is the identity of a particular compound. These peaks can predict the material's crystalline size and atomic arrangement. Identification of crystalline nanoparticle samples is based on adjusting the position of the measured diffraction peaks with the resulting database (peak list).

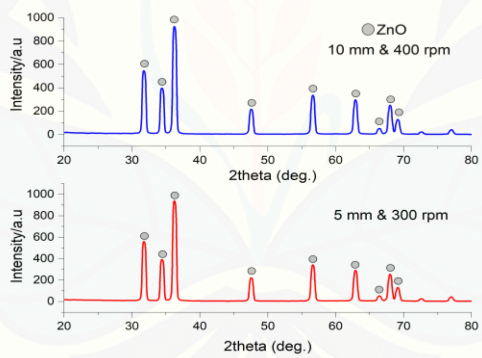


Figure 8. Comparison of XRD parameters of a grinding ball diameter of 5 mm with a disk rotation speed of 300 rpm and a grinding ball diameter of 10 mm with a disk rotation speed of 400 rpm

The results of the XRD test results for the 5 mm diameter grinding ball with a disk rotation speed of 300 rpm and the 10 mm diameter grinding ball with a disk rotation speed of 400 rpm using X-ray CuKa waves (λ = 0.1541874 nm). It can be seen in Figure 8 that the peaks produced in the grinding ball diameter parameter are 5 mm with a disk rotation speed of 300 rpm, while the grinding ball diameter parameter is 10 mm with a disk rotation speed of 400 rpm at angles of 2θ = 31.90, 34.55 and 36, 38 with Miller's Index (hkl) at the three most intense peaks being (100), (002), and (101). The data in the figure matches the ICDD ZnO data standard No. 23, 647-654 on COD (Crystallography Open Database).

The particle size is obtained by calculating the diffraction peak using the Deybe Scherrer equation [2]. The crystal size was obtained with an average of 32.1 nm. In the grinding ball diameter parameter of 10 mm with a disk rotation speed of 400 rpm, the particle size was obtained with an average of 36.34 nm. Very small crystals produce very wide diffraction peaks. So that FWHM affects the crystal size, the greater the

FWHM value, the smaller the crystal size obtained [10]. Crystals are part of the particle, so the size of the crystal is smaller than the particle [15].

The lattice parameters can be obtained using equations (5) and (6). It was obtained at the diameter parameters of 5 and 300 rpm the lattice parameters a = 3.25 and c = 5.2 and at the diameter parameters 10 and 400 rpm the lattice parameters a = 3.25 and c = 5.2. The lattice parameter values obtained are in good agreement with bulk ZnO which has constants a = 3.25 and c = 5.2 [5].

The results of the calculation of the lattice parameters of the ZnO particle crystal structure, then carried out a simulation to describe the formation of Zn and O atoms using Vesta-Student Version software.

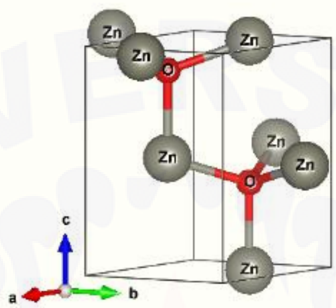


Figure 9. Zno Crystal Formation With VESTA Application

The results of the illustration using Vesta-Student Version software are shown in Figure 9, it can be seen that the ZnO crystal structure formed is hexagonal and the gray ball is a Zn atom and the red one is an O atom, where each O ion is surrounded by four Zn ions. The results of the experimental data illustration describe the hexagonal wurtzite crystal structure in accordance with the results of the study [8]. Hexsagonal wurtzite is a more stable structure than zicblade and rocksalt. Hexsagonal wurtzite is the most stable structure in space compared to other structures that can only be stable under certain conditions and with separate treatment [3]. Another advantage of hexagonal wurtzite is that it has a wider cross-sectional area and is denser so that the surface area is wider [22].

5. CONCLUSION

The particle size parameter for grinding ball 5 with a disk rotation speed of 300 rpm is 61.94 nm and for grinding ball 10 with a disk rotation speed of 300 rpm it is 70.73 nm and for grinding ball 5 with a disk rotation speed of 400 rpm, the particle size is 56, 88 nm and grinding ball 10 with a disk rotation speed of 400 rpm 64.96 nm. The crystal size of the 5 mm grinding ball parameter with a disk rotation speed of 300 rpm got a size of 39.41 nm while the 10 mm grinding ball parameter with a disk rotation speed of 400 rpm got a size of 42.73 nm. The two parameters got the lattice parameter values a = 3.25 and c = 5.2 and the shape of the hexagonal wurtzite structure.

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