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Preparation and Surface Modification ZnO Nano-Powder with Antifriction Properties

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In this work, the surface modified ZnO nano-powder is prepared by the method of the combination of direct precipitation and *in situ* surface modification. The prepared samples are characterized by the means of laser particle size analysis, scanning electron microscopy, X-ray diffraction analysis and Fourier transform infrared spectroscopy. In the tribological test, by adding the surface modified ZnO nano-powder, the friction coefficient and the wear scar diameter of lubricants can be significantly reduced. The powder plays a role in antifriction.

Key Words: Precipitation method, *In situ* surface modification, ZnO nano-powder, Antifriction.

INTRODUCTION

The friction and wear of the problems commonly were existed in any machine. Especially, the large-scale, precision machinery and equipment, it's parts in the lubrication and antiwear performance requirements improve, this will force us to study new additive of lubricating oil[1]. In recent years, research of nanometer materials in the lubrication antifriction and antiwear were rapidly developed. Adding lubricating oil additives have been used in ships, tanks and armored vehicles to improve engine performance and reduce the equipment between parts wear and prolong the service life of the mechanical equipment in the United States and Russia and other countries[2]. Researchers were working on antiwear and antifriction mechanism of lubricating oil with nanometer additives[3]. Yunhui[4] developed nano tin lubricant, in short time internal friction surface get thicker coating, rise to wear self repair function. Guichang *et al.*[5] developed styrene and methacrylic acid, titanium dioxide nanometer microspheres, which has good tribological properties. The domestic research institutions successively in nanometer lubricating oil additive on the research and application and in the military equipment and aerospace and other fields have achieved good effect[6].

EXPERIMENTAL

Preparation of ZnO nano-powder: Firstly, a certain amount of silane coupling agent KH560 dissolved into alcohol solution for hydrolysis (solution A). And a certain amount of polyvinyl dissolved into deionized water and heated in the muffle furnace to 90 ºC until the solution became colourless clear (solution B). To prepare $Zn(NO_3)_2$ solution, a certain amount of $Zn(NO_3)_2$ was weighed and dissolved in deionized water and heated on a magnetic stirrer at 65 ºC until the solution became colourless (solution C). The polyvinyl alcohol solution was selected as a dispersant. After solution B was put into solution C for full dispersion, then solution A was put into and was heated on a magnetic stirrer at 60 ºC. At the same time, the solution was slowly dropped into ammonia water, was controlled reaction temperature 60 ºC and the pH of the solution was *ca.* 8, till no precipitation existed in the solution. After washing by deionized water until no NO_3^-, the precipitate was placed in a vacuum drying box for drying. The dried precipitate was removed by grinding for differential thermal analysis. According to the results of DTA, the calcining temperature of nano-powder was determined.

Characterizations of ZnO Nano-powder: The crystal form of ZnO nano-powder was analyzed by X-ray diffraction analysis. The formation of covalent bands was testified by Fourier Transform Infrared Spectroscopy (FTIR). The diameters of ZnO nano-powder was analyzed by the means of laser particle size analysis (PSA). Through scanning electron microscopy (SEM) was investigated the dispensability of ZnO nano-powder. Finally, the antifriction properties of ZnO nano-powder as additives into lubricating oil were tested by the four-ball wear test.

Friction test

Preparation of samples: The lubricating oil was tested as a sample A. The sample B was prepared that ZnO nano-powder was added into lubricating oil with mass concentration

0.05 %. The oil solution was dispersed in the ultrasonic for 2 h. After ZnO nano-powder was homogenously dispersed in the lubricating oil, the oil solution was kept at room temperature for 3 day before friction tests. The same method, the sample C, the sample D and the sample E were prepared that ZnO nano-powder was Added into lubricating oil with different mass concentration 0.1, 0.5 and 1 %.

Four-ball wear test: This test method for determination of the coefficient of friction of lubricants using the four-ball wear test machine (ANSI/ASTM D5183-2005). In the friction process, test parameters were set as follows, the temperature for: 75 ºC, the load for: 147 n, the speed for: 1450 rpm, the test time for 0.5 h. The samples were four-ball test, the computer automatic recording data.

RESULTS AND DISCUSSION

DTA-TG analysis: Fig. 1 is the DTA-TG results of synthesized ZnO nano-powder in the range of room temperature to 800 ºC. The weight loss reached over 82.5 % before 400 ºC, indicating the ZnO nano-powder mainly converted to oxides at 400 ºC precipitate and the mass loss was mainly caused by the decomposition of the evaporation of the adsorbed water in the $Zn(OH)_2$. In the DTA curve, there was a endothermic peak that appeared at *ca.* 200 ºC was due to the loss of adsorbed water and crystallization water in the precursor.

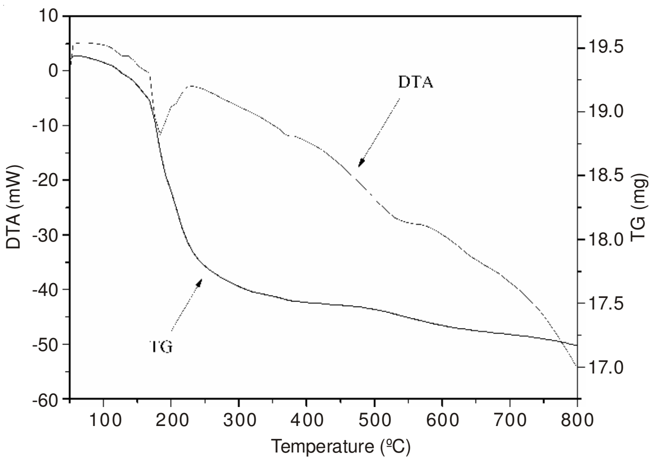


Fig. 1. DTA curve and TG curve of synthesized ZnO nano- powder

XRD analysis: The XRD pattern of synthesized nanopowder after surface modification and calcinations was illustrated in Fig. 2. It can be indexed as orthorhombic crystalline structure with the diffraction peaks d1 = 2.48 Å, d2 = 2.81 Å and d3 = 2.60 Å, which is agreeable with the standard diffraction chart of ZnO in JCPDS-65-3411 indicating the ZnO phase has good crystallinity and there is no impurities.

FTIR analysis: Fig. 3 shows FTIR spectra of ZnO nanopowder (a) and ZnO nano-powder modified with KH-560 (b). For the spectra (b), the pack at 2908 and 2908 cm^{-1} attributed to hydroxyl (-OH) stretching vibration were observed. The absorption peak at 1632 and 1039 cm^{-1} corresponds to (-C=C-). Since peaks in spectra of ZnO nano powder (a) were not appeared, ZnO nano-powder surface modified with KH-560 have (-OH) and (-C=C-).

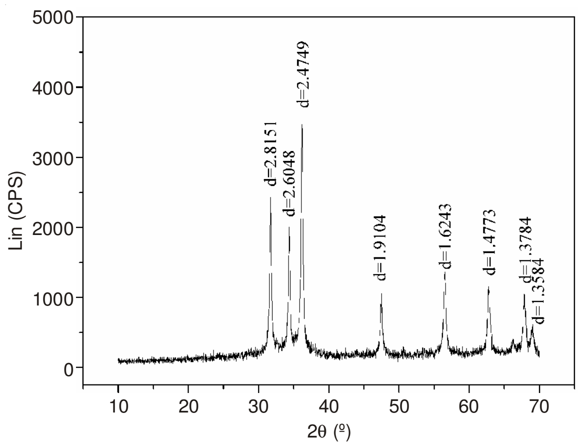


Fig. 2. XRD pattern of ZnO nano-powder after calcination

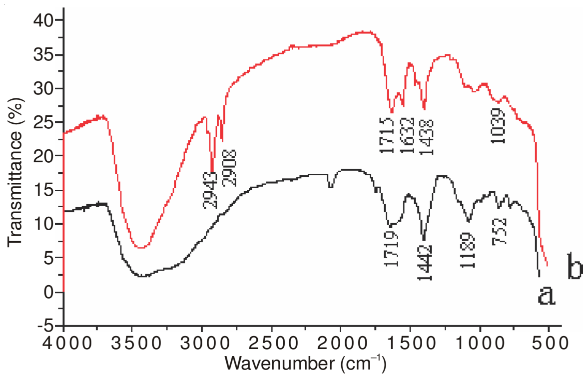


Fig. 3. FTIR spectra of ZnO nano-powder (a) without surface modification (b) with surface modification

Grading analysis: Fig. 4 shows the grading analysis result of synthesized ZnO nano-powder. It can be seen that the particle size of the ZnO samples agreed with normal distribution. The minimum particle diameter was less 40 nm and the average particle diameter was *ca.* 90 nm. The median diameter of particles was approximately 76 nm.

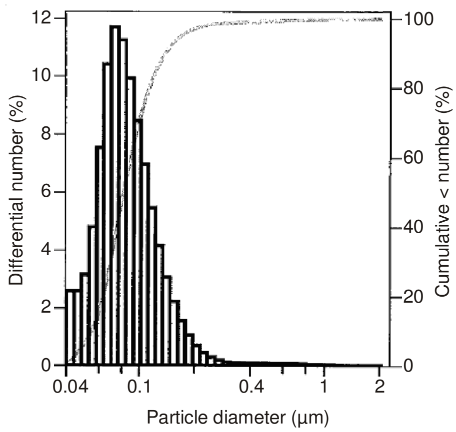


Fig. 4. Grading analysis result of synthesized ZnO nano-powder

Table-1 lists the size distribution of ZnO nano-powder. It can be seen that the particles less 53, 61 and 76 nmwere nearly 10, 25 and 50 % in the ZnO nano-powder, respectively. The particle less 109 nm was nearly 75 %, indicating the size distribution of particle was well and the synthesized powder was nano size.

TABLE-1
GRADING ANALYSIS TABLE OF SYNTHESIZED ZnO NANO-POWDER

> 10 %	> 25 %	> 50 %	> 75 %	> 90 %
0.053 µm	0.061 µm	0.076 µm	0.109 µm	0.131 µm

SEM analysis: SEM images (Fig. 5) shows of ZnO nano-powder. It can be seen that the most of the particles of calcined ZnO powder was relatively small and distributed well. The morphology of particle was regular and nearly spherical. Only a small part of particles agglomerated.

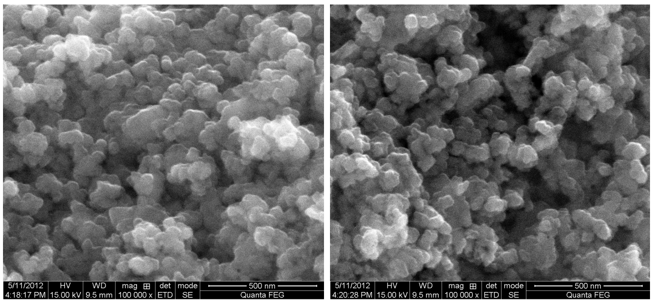
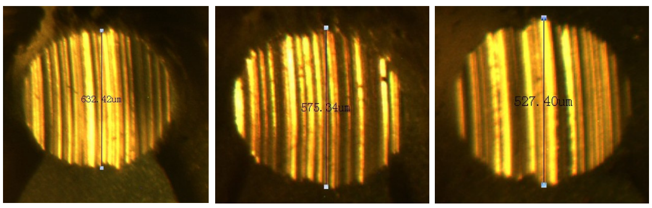
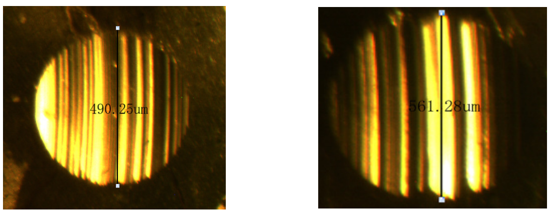


Fig. 5. SEM images of ZnO nano-powder

Friction tests analysis: Fig. 6 shows wear scars of samples by four-ball wear tests. The antiwear properties were changed because modified ZnO nano-powder joined into lubricating oil. The wear scars diameter of the sample D (concentration 0.5 %) by four-ball wear tests is 490.25 µm and sample D's is 632.42 µm. Fig. 7 showed friction coefficients changes of samples by four-ball wear tests. Friction coefficients of samples decreased with concentrations of ZnO increased until the concentration was 0.5 % and concentrations of ZnO increased friction coefficients of samples increased. Table-2 shows wear scars diameters and average friction coefficient by four ball wear tests. At 0.5 % concentrations of ZnO, the friction coefficient of sample decreased to minimum value, the sample D antifriction the best performance.



(a) Sample A (b) Sample B (c) Sample C



(d) Sample D (e) Sample E

Fig. 6. Wear scars of samples by four-ball wear tests

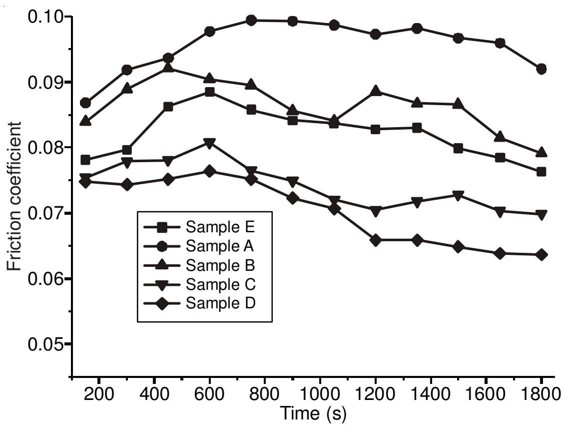


Fig. 7. Friction coefficients of samples by four-ball wear tests

TABLE-2
RESULTS OF FOUR-BALL WEAR TESTS

No	Wear scar diameter (µm)	Friction coefficients
Sample A	632.42	0.096
Sample B	575.43	0.087
Sample C	527.40	0.076
Sample D	490.25	0.071
Sample E	561.28	0.083

Conclusion

• ZnO nano-powder was synthesized by direct precipitation and *in situ* surface modification. The minimum size of particle was less than 40 nm and the average diameter of particle of was *ca.* 90 nm. The median diameter of spherical particles was *ca.* 76 nm.

• Hydrolysis of silane coupling agent (KH-560) can well ZnO nano-powder surface modified with (-OH) and (-C=C-). After ZnO nano-powder was achieved the effect of surface modification, it was well dispersion and stability in the lubricating oil.

• Through four-ball wear tests were concluded that ZnO nano-powder additive to put into lubricating oil were played a better antiwear effect, the friction coefficient and the wear scar diameter can be significantly reduced. When the ZnO nano-powder in lubricating oil concentration was 0.5 %, produced the best antiwear effect.

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