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Preparation and Surface Modification SiO_2 Nano-Powder with Antifriction Properties

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In this paper, ethyl silicate as the main raw material, SiO_2 was prepared by precipitation method. The powder was *in situ* surface modification, get a small particle size and stability in the lubricating oil, monodisperse spherical SiO_2 nanoparticles. Using X-ray diffraction analysis, infrared spectroscopy, laser particle size analysis, scanning electron microscopy and other methods to characterize them. The study results showed that the optimal solution to preparate of SiO_2-*situ* surface modification of nano-powder is ethanol as solvent, KH-560 as a surface modifier, the holding time of 3 h, the temperature was 80 °C. The resulting average particle size of SiO_2 nanoparticles is 91 nm and uniform, as regular sphere. It can be obtained by the infrared spectrum that KH-560 bonded with the SiO_2 nanoparticles *via* a covalent bond, organic coating layer formed on the surface, the SiO_2 nanoparticles changes from hydrophilic oleophobic to lipophilic hydrophobic. The amount of the powder added to lubricating oil as anti-wear agents, the long grind four-ball friction test, study its antifriction properties. The results showed that SiO_2 nanoparticles are added to the lubricating oil has obvious antifriction effect, and the optimal dosage is 0.1 wt %.

Keywords: Chemical precipitation, SiO_2 nanoparticles, Antifriction properties.

INTRODUCTION

Silicon dioxide (SiO_2) nano powder is prepared by the gas phase and liquid phase method. use of gas phase method, high purity SiO_2, dispersion is very good, very small particle size, but particularly high production particularly large energy consumption and cost [1]. Liquid prepared nanoparticles because of its relevant industrial process control and equipment of amplification technology is relatively mature, the preparation of nanoparticles preparation technology of the most competitive advantage [2]. Common methods include sol-gel method, hydrothermal method, precipitation, alcohol saline solution [3]. With precipitation of SiO_2 nano powder surface modification method modification and coupling agent modification [4].

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 anti-wear performance requirements improve, this will force us to study new additive of lubricating oil [5]. Researchers were working on anti-wear and antifriction mechanism of lubricating oil with nanometer additives [6]. Yunhui *et al.* [7] developed nano tin lubricant, in short time internal friction surface get thicker coating, rise to wear self repair function. Jiang *et al.* [8] 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 [9].

EXPERIMENTAL

Preparation of SiO_2 nano-powder: Firstly, a certain amount of silane coupling agent KH560 dissolved into alcohol solution for hydrolysis (solution A). A certain amount of polyvinyl alcohol dissolved into deionized water and heated in the muffle furnace to 90 °C until the solution became colourless clear (solution B). For preparation ethyl silicate solution, a certain amount of ethyl silicate was weighed and dissolved in deionized water and heated on a magnetic stirrer at 60 °C 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 °C. At the same time, the solution was slowly dropped into ammonia water, was controlled reaction temperature 60 °C till no precipitation existed in the solution. The precipitate was placed in a vacuum drying box for drying.

Characterizations of SiO_2 nano-powder: The crystal form of SiO_2 nano-powder was analyzed by X-ray diffraction analysis (XRD). The formation of covalent bands was testified by Fourier transform infrared spectroscopy. The diameters of SiO_2 Nano-powder was analyzed by the means of laser particle size analysis. Through scanning electron microscopy was investigated the dispensability of SiO_2 nano-powder. Finally, the antifriction properties of SiO_2 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 SiO_2 nano-powder was added into lubricating oil with mass concentration 0.05 %. The oil solution was dispersed in the ultrasonic for 2 h. After SiO_2 nano-powder was homogenously dispersed in the lubricating oil, the oil solution was kept at room temperature for 3 days before friction tests. The same method, the sample C, the sample D and the sample E were prepared that SiO_2 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 °C, the load for: 147 n, the speed for: 1450 rpm, the test time for 30 min. The samples were four-ball test, the computer automatic recording data.

RESULTS AND DISCUSSION

The XRD pattern of synthesized nano-powder after surface modification and calcinations was illustrated in Fig. 1.

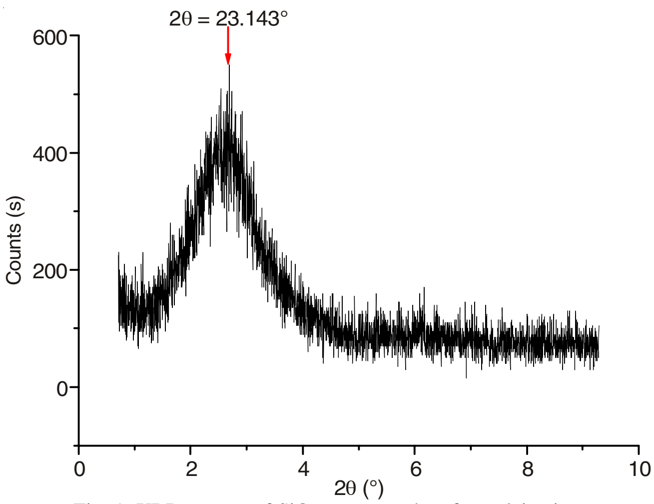


Fig. 1. XRD pattern of SiO_2 nano-powder after calcination

Fig. 2 shows FTIR spectra of SiO_2 nano-powder (a) and SiO_2 nano-powder modified with KH-560 (b). For the spectra (b), the pack at 2908 cm^{-1} attributed to hydroxyl (-OH) stretching vibration were observed.

Grading analysis: Fig. 3 shows the grading analysis result of synthesized SiO_2 nano-powder. It can be seen that the particle size of the SiO_2 samples agreed with normal distribution. The minimum particle diameter was less than 40 nm and the average particle diameter was about 91 nm. The median diameter of particles was approximately 80 nm.

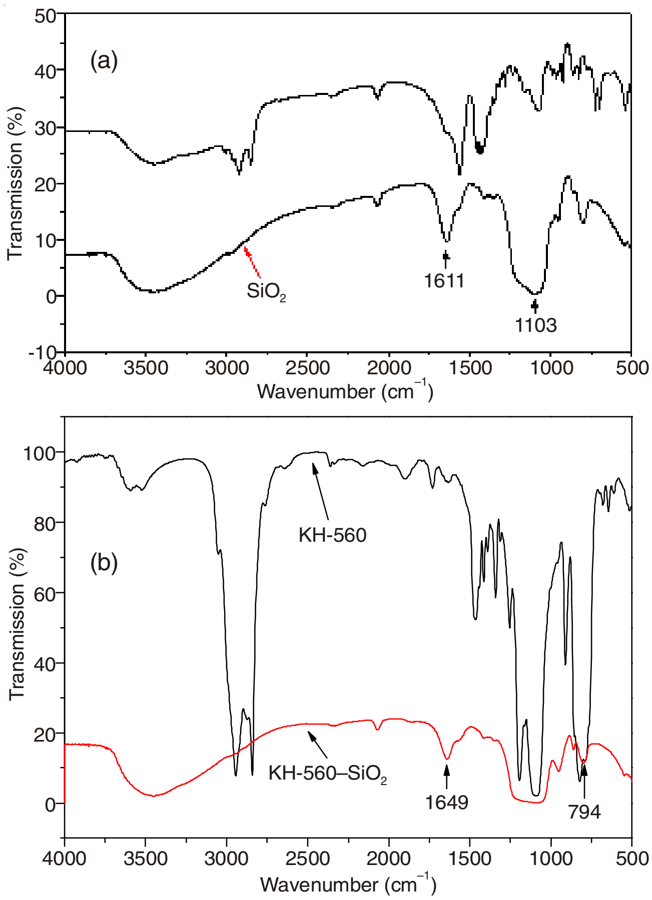


Fig. 2. FTIR spectra of SiO_2 nano-powder (a) without surface modification (b) with surface modification

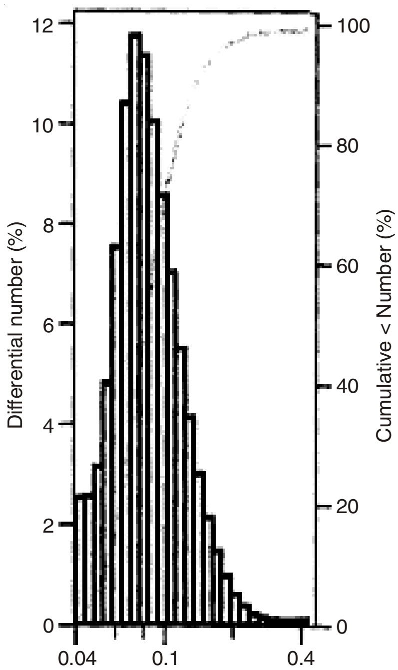


Fig. 3. Grading analysis result of synthesized SiO_2 nano-powder

SEM analysis: Fig. 4 SEM images shows of SiO_2 nano-powder. It can be seen that the most of the particles of calcined SiO_2 powder was relatively small and distributed well. The morphology of particle was regular and nearly spherical. Only a small part of particles agglomerated.

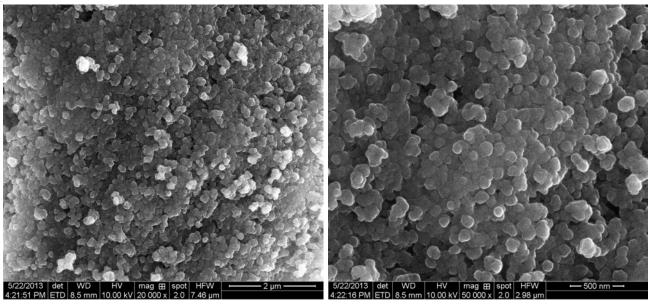
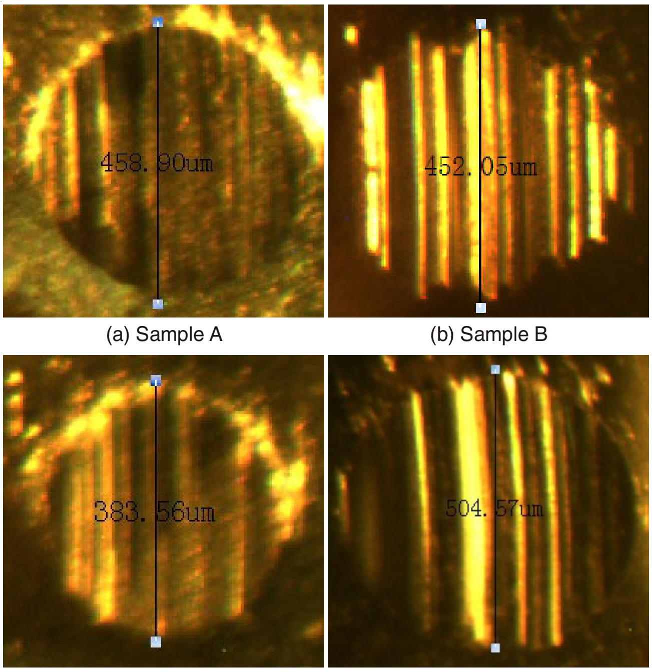


Fig. 4. SEM images of SiO_2 nano-powder

Friction tests analysis: Fig. 5 shows wear scars of samples by four-ball wear tests. The anti-wear properties were changed because modified SiO_2 nano-powder joined into lubricating oil. The wear scars diameter of the sample C (concentration 0.1 %) by four-ball wear tests is 383.56 μm and sample D's is 504.57 μm. Fig. 6 showed friction coefficients changes of samples by four-ball wear tests. Friction coefficients of samples decreased with concentrations of SiO_2 increased until the concentration was 0.1 % and concentrations of SiO_2 increased friction coefficients of samples increased.



(a) Sample A (b) Sample B

(c) Sample C (d) Sample D

Fig. 5. Wear scars of samples by four-ball wear tests

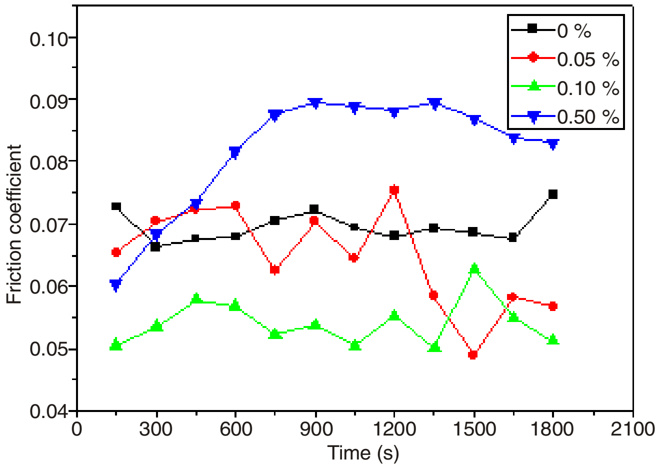


Fig. 6. Friction coefficients of samples by four-ball wear tests

Conclusion

• Silicon dioxide (SiO_2) nano-powder was prepared 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 about 91 nm. The median diameter of spherical particles was approximately 80 nm.

• Through four-ball wear tests were concluded that SiO_2 nano-powder additive to put into lubricating oil played a better anti-wear effect, the friction coefficient and the wear scar diameter can be significantly reduced. When SiO_2 nanoparticles are added to the lubricating oil has obvious antifriction effect. The optimal dosage is 0.1 wt %.

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