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TRIBOLOGICAL STUDIES ON NANO-CaCO3 ADDITIVE MIXED LUBRICANT

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ABSTRACT: *$CaCO_3$ nanoparticles with an average size of 285 nm were synthesized via the carbonation method. The tribological properties of the $CaCO_3$ nanoparticles as an additive in lithium grease were evaluated with a four-ball tester. The results show that these $CaCO_3$ nanoparticles exhibit good performance in anti-wear and friction-reduction, load-carrying capacity, and extreme pressure properties. The action mechanism was estimated through analysis of the worn surface with scanning electron microscope (SEM). The results indicate that a boundary film mainly composed of $CaCO_3$, CaO, iron oxide, and other organic compounds was formed on the worn surface during the friction process.*

Keywords: *$CaCO_3$ nanoparticles, Lubricant additives, Nanotribology, Lithium grease.*

I. INTRODUCTION:

The Nano particles are used in various research fields, because of their special physical and chemical properties. In this paper nanoparticles were prepared by carbonation method for the calcium carbonate nanoparticles. Significantly improve its anti-wear property, load carrying capacity and friction reduction property[1]. Complicated tribochemical reaction occurred in boundary lubrication process[2]. The optimum concentration of nanoparticle additive in lithium grease is greatly improve their anti-wear and extreme pressure property[3]. Tribological performance of the two lubricating grease under the boundary lubrication condition were investigated at the time grease showed excellent friction-reducing and anti wear properties regardless of the testing applied load and frequency[4]. The tribo chemical reactions of boundary tribofilms consisted and organic compounds were formed on the rubbing surfaces[5]. Different additives at optimal concentrations have no effect on increasing base grease load-carrying capacity. The main wear mechanism on wear scar is slight adhesive wear[6].

II. EXPERIMENTAL:

2.1 Synthesis of $CaCO_3$ nanoparticles:

The nanoparticles of $CaCO_3$ were prepared by the carbonation method[1]. The 5wt% of calcium oxide is mixed into the distilled water at the temperature of 80º and kept at 24hrs. Then, carbon dioxide gas carried into the solution at the temperature of 16ºc and reached the pH level in 7. The solution was monitored by the pH meter. The white precipitate was washed with deionized water and ethanol several times. Finally the product was dried in a vaccum oven at 60ºc for 24 hr.

Lithium grease:

The multipurpose lithium grease was purchased and to make nano grease. The effect of $CaCO_3$ nanoparticles additives on the tribological behavior of lithium grease, $CaCO_3$ nanoparticles were added into lithium grease at the different concentrations, mixed by mechanical stirring and grinding for three times in triple-roller mill.

2.2 Friction and Wear Tests

The tribological behavior of $CaCO_3$ used as an additive in lithium grease was tested on a MRS-1J four-ball tester (Jinan, Fig. 1). The friction and wear tests were performed at a rotating rate of 1,200 rpm (linear speed of 33.38 m/min), a load of 100, 200, 300, 400 and 500 N, room temperature and for test duration of 60 min. The peak/average Hertz pressures at normal loads of 100, 200, 300, 400 and 500 N were calculated to be 2.177/1.452 GPa, 2.743/1.829 GPa, 3.140/2.093 GPa, 3.456/2.304 GPa and 3.723/2.482 GPa, respectively. The maximum non-seizure loads were obtained by a MRS-10 (G) four-ball tester (Jinan instrument manufacturer, PR China) according to the national standard GB/T3142-82, which is similar to the ASTM D2783. The test balls ([12.7 mm, HRC 60 ± 1) were made of GCr15. The wear scar diameters (WSD) of the three lower balls were measured on a digital-reading optical microscope to an accuracy of ±0.01 mm, and the average wear diameter of the three lower balls was calculated as the WSD in this experiment. All tests were performed three times for each sample and the friction coefficients were recorded automatically by the computer with a data-acquiring system linked to the four-ball tester.

2.3 Characterization

Powder X-ray diffractometer (RigaKu D/max- RB, Japan) using Cu Ka radiation (k = 1.54056 A ° , 40 kV, 30 mA) was used to identify the phase of $CaCO_3$ nanoparticles. The worn surfaces of the lower steel balls were investigated by a JEOL 6390 scanning electron microscope (SEM) equipped with energy dispersive spectra (EDS). The upper steel balls were analyzed by a PHI-5702 X-ray photoelectron energy spectrometer using MgKa radiation as the exciting source and the binding energy of the contaminated carbon (C1 s: 284.6 eV) as the reference, at a pass energy of 29.35 eV and a resolution of about ±0.3 eV.

Fig. 1 Four ball wear test

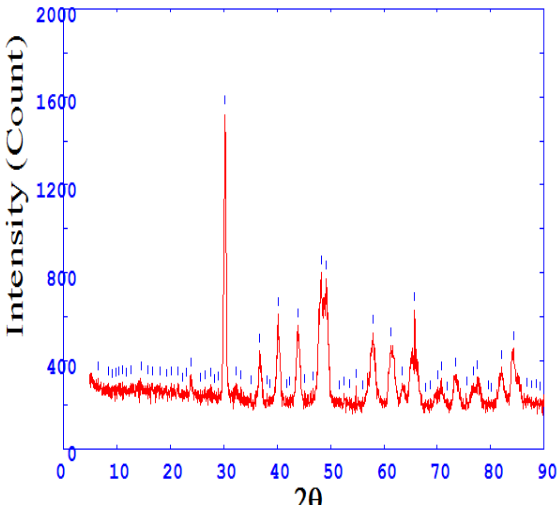


Fig. 2 XRD pattern of CaCO3 nanoparticles

III. Results and Discussion

3.1 Characterization of $CaCO_3$ Nanoparticles

The XRD pattern of the synthesized $CaCO_3$ nanoparticles in Fig. 2 shows that the diffraction peaks match well with literature patterns (JCPDF Card no. 04-0636), indicating that the phase of the $CaCO_3$ nanoparticles is calcite. The average size of the $CaCO_3$ nanoparticles is estimated by the Debye–Scherrer formula to be 280–290 nm. The average size of the nanoparticles is about 285 nm, which is in good agreement with the calculated result from the XRD pattern.

Nano Zeta sizer

The particle size in nanometer range and dispersion stability of ultrafine particles in nanofluids was measured by nano zeta sizer(Model: Nano ZS90, Malvern) is show in figure 3. The sample was prepared by dispersing small amount of ultrafine particles in deionized water with constant ultrasonication and magnetic stirring for 30 minutes each. Then the sample was kept in a sample holder with the help of syringe and analyzed.

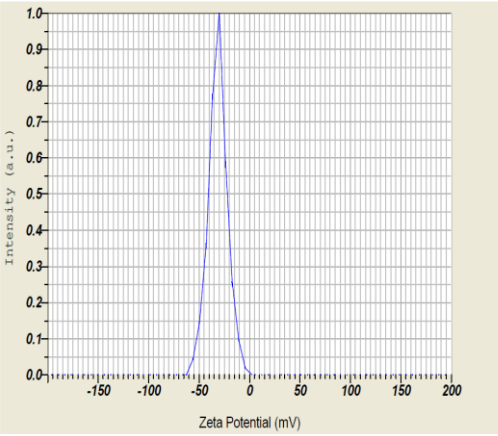


Fig. 3 Zeta Potential graph

CaCo3.nzt
Measurement Results

Measurement Type : Zeta Potential
Sample Name : CaCo3
Temperature of the holder : 25.0 °C
Viscosity of the dispersion medium : 0.896 mPa·s
Conductivity : 0.088 mS/cm
Electrode Voltage : 3.9 V

Calculation Results

Peak No. 1	-31.3 mV	-0.000242 cm2/Vs
2	--- mV	--- cm2/Vs
3	--- mV	--- cm2/Vs

Zeta Potential (Mean) : -31.3 mV
Electrophoretic Mobility mean : -0.000242 cm2/Vs

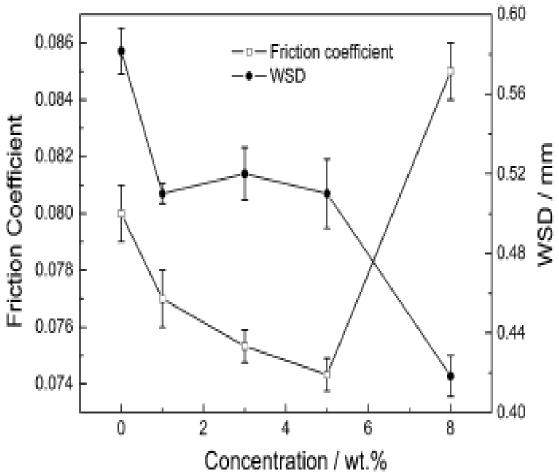


Fig.4 Friction coefficient and wear scar diameter as a function of $CaCO_3$ concentration (four ball, 1200 rpm, 400 N, 60 min.

3.2 Tribological Properties of $CaCO_3$ Nanoparticles

Figure 4 gives the tribological behavior as a function of the additive concentration of CaCO3 nanoparticles under a load of 400 N. It can be seen that the friction coefficient of lithium grease containing $CaCO_3$ nanoparticles decrease gradually with the increase of the $CaCO_3$ nanoparticles concentration in the range of 1–5 wt.%, and then increase rapidly with the $CaCO_3$ nanoparticles concentration at 8 wt.%. While, the WSDs of the lithium grease containing $CaCO_3$ nanoparticles are much lower than that of pure lithium grease at all concentrations. The more the $CaCO_3$ nanoparticles concentration in lithium grease the lower is the corresponding WSD. That is to say, these $CaCO_3$ nanoparticles could improve the anti-wear properties of lithium grease. In our opinion, $CaCO_3$ nanoparticles may be deposited on the rubbing surfaces forming a boundary film during the friction process. When the $CaCO_3$ nanoparticles concentration reached 8 wt.%, thicker films were formed which reduce wear. Thus, the WSD of the lithium grease containing 8 wt.% $CaCO_3$ nanoparticles is much lower. However, thicker boundary film thickness may lead to larger real contact area, which increases friction coefficient. Therefore, the optimum concentration of the $CaCO_3$ nanoparticles as an additive in lithium grease was suggested to be 5 wt.%.

Table 1 The maximum non-seizure load (P_B value) and sintered load (P_D value) of pure lithium grease and 5 wt.% $CaCO_3$/lithium grease

	Lithium grease	$CaCO_3$
P_B (N)	470	549
P_D (N)	1,961	3,089

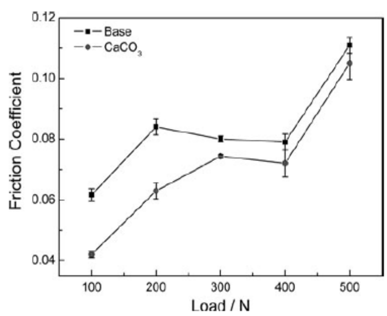


Fig. 5 Friction coefficient as a function of applied load with the lubrication of lithium grease alone and that containing 5 wt.% $CaCO_3$ nanoparticles (four-ball, 1,200 rpm, 60 min)

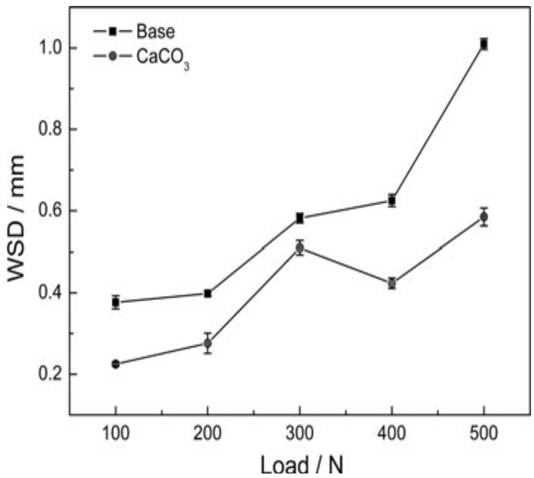


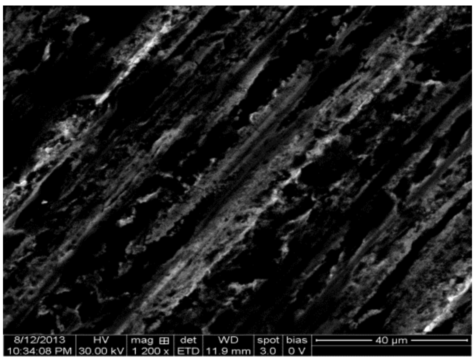
Fig. 6 Wear scar diameter as a function of applied load with the lubrication of lithium grease alone and that containing 5 wt.% $CaCO_3$nanoparticles (four-ball, 1,200 rpm, 60 min)

Figures 5 and 6 give the friction coefficient and WSD as a function of applied load with the lubrication of lithium grease alone and that containing 5 wt.% $CaCO_3$ nanoparticles. From Figs. 5 and 6, it can be seen that the friction coefficient and WSD have the similar trend of variation with load. The friction coefficient and WSD of base grease and the grease containing 5 wt.% $CaCO_3$ nanoparticles become larger with the increase in applied load. In addition, the friction coefficient and WSD of the grease containing 5 wt.% $CaCO_3$ nanoparticles is much smaller than that of the steel ball lubricated by the lithium grease alone, indicating that the $CaCO_3$ nanoparticles as an additive are effective in improving the friction-reduction properties and anti-wear ability of base grease.

3.3 SEM Analysis of Worn Surfaces

In order to investigate the lubrication mechanism of $CaCO_3$ nanoparticles, SEM was used to investigate the worn surface. Figure 7 gives the typical SEM images of the worn steel ball surface lubricated by lithium grease and the grease containing 5 wt.% $CaCO_3$ nanoparticles at 400 N for 60 min. It can be seen that the surface lubricated by lithium grease alone is rough and shows signs of severe scuffing (Fig. 7a). On the other hand, scuffing on the surface lubricated by lithium grease containing 5 wt.% $CaCO_3$ nanoparticles was significantly inhibited (Fig. 7b). Moreover, the wear scar of the steel ball lubricated by lithium grease containing 5 wt.% $CaCO_3$ nanoparticles is much smoother and smaller than that lubricated by lithium grease alone. Therefore, the results further testify that $CaCO_3$ nanoparticles have good anti-wear property.

Figure 8 shows the EDS spectrum obtained from the worn scar on the steel balls lubricated by lithium grease containing 5 wt.% $CaCO_3$ nanoparticles at 300 N for 30 min. It can be seen that the element of Ca was present on the worn scar surface and its atomic concentration was 3.17%. This fact shows that Ca in $CaCO_3$ nanoparticles was deposited on the worn steel surface in the process of friction. The presence of Ca gives strong evidence that a lubricate film must be formed and probably contains $CaCO_3$ nanoparticles, which can prevent the steel-to-steel direct contact.



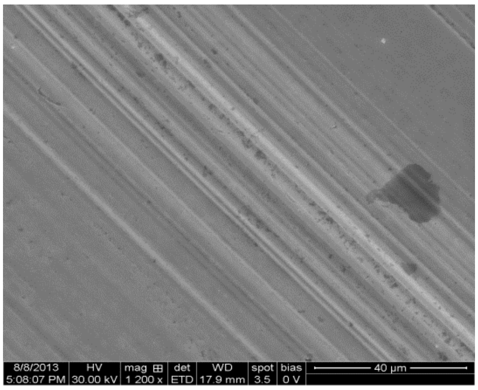


Fig. 7 SEM morphologies of wear scar of steel balls lubricated by **a** lithium grease and **b** 5 wt.% CaCO3/lithium grease at 400 N for 60 min.

It can be concluded that the $CaCO_3$ nanoparticles contained in the lithium grease are trapped inside the contact and fill in the gap between the rubbing surfaces. Under mild test conditions, the nanoparticles facilitated reduction of friction and wear. Under boundary lubrication conditions, the nanoparticles were deposited on the shearing surface because of the high surface energy of the fresh wear surface and a complicated tribochemical reaction that had occurred. The tribochemical reaction film is composed of absorbed organic materials coming from additives or lithium grease itself, CaO, and iron oxide, which act as solid lubricants during the friction process, resulting in good tribological behavior on the rubbing surface of the friction pair. Therefore, the deposits of $CaCO_3$ nanoparticles and tribochemical reaction products on the worn surface improve the tribological properties of lithium grease.

IV. Conclusions

According to the results and discussion above, the following conclusions can be drawn:

1. $CaCO_3$ nanoparticles were successfully prepared via a carbonation method. The synthesized cubic $CaCO_3$ nanoparticles have an average diameter of 285 nm and could be well dispersed in lithium hydroxystearate (soap) fiber, when added in lithium grease.
2. $CaCO_3$ nanoparticles as an additive in lithium grease significantly improve its anti-wear performance, friction- reduction property, load-carrying capacity, and extreme pressure property. The optimum concentration is 5 wt.%.
3. During the friction process, $CaCO_3$ nanoparticles were deposited on the rubbing surfaces forming a boundary film. A complicated tribochemical reaction also occurred in the boundary lubrication process. From EDS analysis of the corresponding worn steel surface, it can be seen that the boundary film was composed of $CaCO_3$ nanoparticles, CaO, iron oxide, and other organic compounds.

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