



Tribological behaviors of surface-coated serpentine ultrafine powders as lubricant additive

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ABSTRACT

The effect of surface-coated ultrafine powders (UFPs) of serpentine suspended in lubricants on the tribological behaviors of a mated 1045 steel contact was investigated. Through the addition of serpentine UFPs to oil, the wear resistance ability was improved and the friction coefficient was decreased. The addition of 1.5 wt% serpentine to oil is found most efficient in reducing friction and wear. The nano-hardness and the ratio of hardness to modulus of friction surface are observably increased. Such effects can be attributed to the formation of a tribofilm of multi-apertured oxide layer, on which the micrometric alumina particles embedded and serpentine nano-particles adsorbed.

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1. Introduction

Over the last few years, interest in the synthesis and tribological properties of ultrafine powders (UFPs) as lubricant additives has steadily grown due to their efficacies in reducing friction and wear [1–4]. There have been many investigations on the behaviors of inorganic or organic UFPs as extreme pressure (EP) and anti-wear (AW) additives for liquid lubricants. It is found that the lubricating properties of oils were significantly improved when nano-sized particles were used, while micro-sized particles had much smaller effects [5–7].

Compared with nano-particles, the micrometric UFPs in liquid media are more thermodynamically unstable and tend to spontaneously subside. The large-scale particles, on the other hand, can act as abrasive particles on contacts, as accordingly result in severe wear. Therefore, the previous and current studies on UFPs additives mainly focused on the tribology testing of nano-scale particles of metals [8–13], carbon materials [14–17], oxides [18–20], sulfides [21–23], borates [24], RE compounds [25–27], polymers [28], etc. Besides BN particles [29,30], few studies were carried out on micro-sized UFPs due to their small contribution towards friction-reducing and wear resistance.

Serpentine group describes a group of common rock-forming hydrous magnesium iron phyllosilicate ((Mg,Fe)₃Si₄O₁₀(OH)₂) minerals. They may contain minor amounts of other elements including chromium, manganese, cobalt and nickel. Recent researches indicate

the micro-sized serpentine (Mg₃[Si₄O₁₀](OH)₂) UFPs present excellent tribological properties when added to liquid lubricants [31,32]. Jin et al. [33] investigated the tribological behaviors of crankcase oil suspended serpentine particles ($\leq 2.0 \mu\text{m}$) in railway diesel engines under field trial conditions. They found that a super-hard and super-lubricious oxide layer formed on the worn ferrous surface, as accordingly lowers the friction and wear. Yu et al. [34] studied the lubricating effect of natural mineral admixtures (size: 0.3–3 μm) that mainly composed of serpentine (90–95%) and schungite (4.8–9.8%). They found that a DLC film with Si or Si–O structures doped formed on the worn steel contacts, as contributes to the excellent mechanical and tribological properties of the friction surface.

The present study aimed to further clarify the mechanisms responsible for the effect of serpentine minerals used as additives. The effect of surface-coated serpentine UFPs, with an average particle size of 1.0 μm , suspended in lubricating oils on the tribological behaviors of steel contact was reported. The morphologies and element distributions of the tribofilm formed by the serpentine were studied. In particular, the nano-mechanical properties of the tribofilm were measured by a nano-indentation tester.

2. Experimental

2.1. Materials

The UFPs used in the present study were prepared by mechanical crushing and ball-milling the serpentine mineral

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Table 1
Chemical composition of the serpentine minerals.

Oxides	Content (wt%)
SiO ₂	43.49
Al ₂ O ₃	1.18
FeO	0.25
MnO	0.32
MgO	41.0
CaO	0.64
K ₂ O	0.33
P ₂ O ₅	0.085
H ₂ O ⁺	12.37
H ₂ O ⁻	0.29

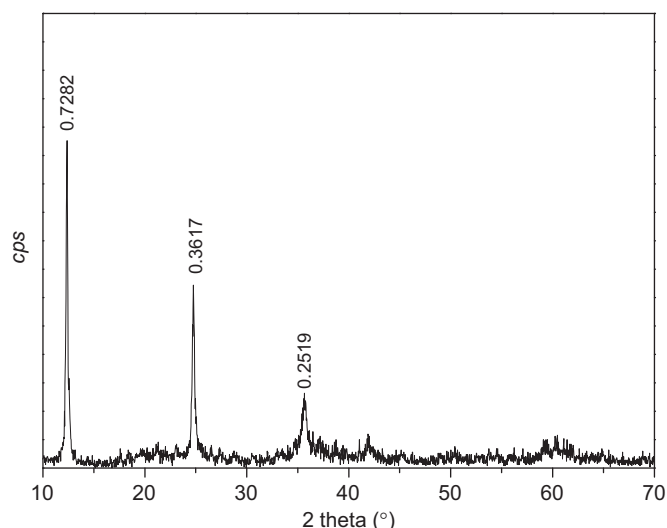


Fig. 1. XRD pattern of untreated serpentine UFPs.

(Liaoning Province, China). Table 1 lists the chemical compositions of the starting materials, its crystal formula can be expressed as $\text{Mg}_{5.70}\text{Al}_{0.13}\text{Fe}_{0.02}\text{Ca}_{0.06}\text{K}_{0.04}\text{Mn}_{0.03}[\text{Si}_{4.05}\text{O}_{10}](\text{OH})_8$, which is close to the ideal formula of serpentine, i.e. $\text{Mg}_6[\text{Si}_4\text{O}_{10}](\text{OH})_8$. Fig. 1 shows the X-ray diffraction pattern of the ball-milled serpentine UFPs. The diffraction peaks of $d=0.7282$, 0.3617 and 0.2519 nm can be indexed to those of antigorite, corresponding to the (001), (102), and (16.0.1) planes, respectively. Its lattice parameters are calculated as follows: $a=0.536$ nm, $b=0.928$ nm, $c=0.732$ nm, $\alpha=\gamma=90^\circ$ and $\beta=91.38^\circ$, further proving the antigorite structure. To provide good stabilization in viscous liquid, a mixture of boric acid ester and Span 60 (mol. ratio=1:1) was mixed with the UFPs in a globe mill for 6 h operation (rotating speed=300 rpm) to produce an organic coating layer. Fig. 2 shows the SEM image and size distribution of the surface-coated serpentine UFPs. The particle size is mostly in the range of $0.1\text{--}5\text{ }\mu\text{m}$ and the average size approximates $1.0\text{ }\mu\text{m}$. The final surface-coated UFPs can be dispersed well in some organic solvents, such as chloroform, benzene, methylbenzene and lubricating oil.

2.2. Friction and wear test

An MM-10W sliding friction tribotester was employed to study the friction-reduction and anti-wear abilities. As shown schematically in Fig. 3, the MM-10W comprises an upper rotating ring specimen, which came into contact with a lower disk specimen fixed in an oil bath. The friction coefficient and temperature of the disk specimen were then measured. Diesel engine oil (grade:

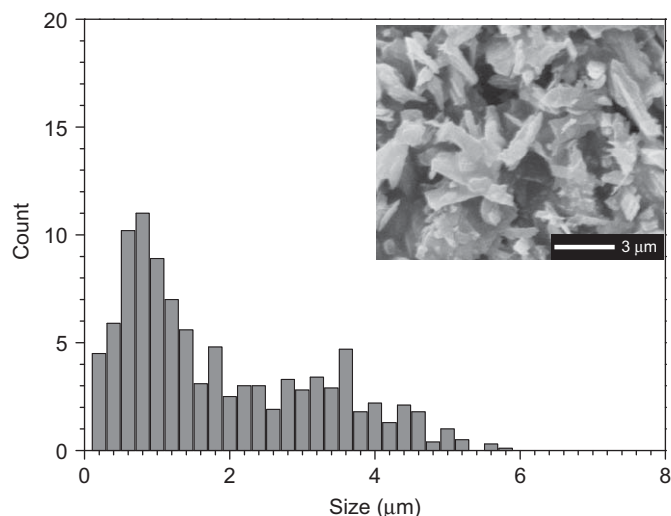


Fig. 2. SEM image and size distribution of surface-coated serpentine UFPs.

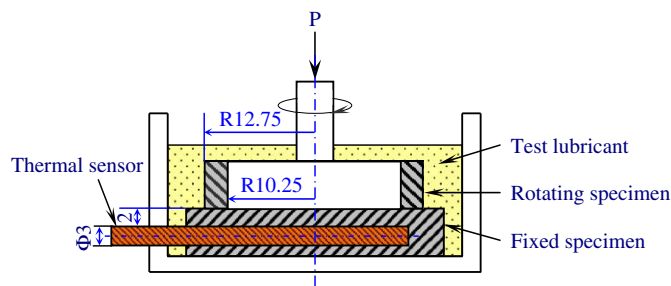


Fig. 3. Schematic illustration of friction and wear test.

Table 2
Viscosity of oil with and without serpentine UFPs.

	Kinematic viscosity (100 °C) (mm ² /s)	Viscosity (150 °C, 10 ⁶ s) (mPa s)
Pure oil	18.6	3.7
0.5 wt% UFPs	18.6	3.7
1.0 wt% UFPs	18.6	3.7
1.5 wt% UFPs	18.7	3.7
2.0 wt% UFPs	18.7	3.8

50CC) was used as a lubricant for the friction specimens of the rings and disks (1045 steel, hardness: 210 HB, surface roughness: $0.20\text{--}0.25\text{ }\mu\text{m}$). Table 2 gives viscosity of the oil with and without serpentine UFPs. High-energy mechanical ball-milling agitation (rotating speed=120 rpm, duration=60 min) and ultrasonic dispersion (power=200 W, temperature=40 °C, duration=30 min) were used to provide good dispersion stability of the surface-coated UFPs in oil. The experimental conditions were: atmospheric environment, room temperature, normal load=100, 200, 300 and 400 N, sliding speed=1.51 m/s and test duration=120 min. The corresponding initial mean Hertzian pressure at the contacts was 0.554 MPa (100 N), 1.11 MPa (200 N), 1.66 MPa (300 N) and 2.21 MPa (400 N). After the test, the steel disks were cleaned in petroleum ether and absolute ethyl alcohol. The wear rates of the disks were then calculated by

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