Surface-Reconditioning Additives Based on Solid Inorganic Nanoparticles for Environment-Friendly Industrial Lubricating Compositions

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Abstract

Our research is aimed at the application of lamellar ceramic solid nanoparticles as surface reconditioning additives to industrial lubricating oils to achieve self-repair and improve lubricity. According to a NERC, lubrication failures are among the top causes of outages and deratings of hydroelectric turbines. This problem represents a tremendous opportunity to improve the reliability and availability of hydroelectric turbines by improving their lubricating technologies. The majority of the environmental toxicity of these lubricating compositions is from the additives, where few alternative options are being explored. Today, the ability to formulate lubricating compositions that are safe for the environment greatly depends on additives. There has been a steadily growing interest toward solid, inorganic nanopowders of natural minerals such as Magnesium Hydro-Silicates(MHS) as antiwear and friction modifying additives in lubricating oils. Such powders can reduce wear and promote the formation of thick (up to 30 microns) tribofilms on the rubbing surfaces with great lubricating properties. Self-regulating mechanism of a film formation, and the ability to compensate for wear, allows for the self-repair effect to be achieved. This research is directed at expanding our understanding of the industrial applications for this technology and not only improve current lubricating compositions, but also note additional effects: such as superlubricity and reconditioning worn surfaces. We evaluated the influence of temperature, pressure, and concentration on friction properties. The optimal concentration of nanoparticles was obtained for steel-on-steel friction pairs. Our additives can be applied toward regular and preventative maintenance in the power generating industries as well as emergency surface treatment after lubrication failure has occured to compensate for wear.

1.0 Introduction

According to a NERC, lubrication failures are among the top causes of outages and deratings of hydroelectric turbines[1].
Methods for minimizing friction losses have a significant importance; mineral or synthetic oil-based lubricants are the most popular solution to friction and wear related losses for many steady regime applications. Under optimal conditions, oils can significantly lower friction losses as well as minimize wear. To minimize losses, rubbing partners must be separated by a thin lubrication film and the speed of the mating parts must match the properties of the lubricating film. Under such conditions, surface asperities become elastic, which is called Elasto-Hydrodynamic Lubrication or EHL. Among many factors, the main parameter to achieve EHL is the viscosity of the lubricant; however, since many mechanisms work during the dynamic operation of moving parts at a variable speed, the optimal regime may not always be achieved using lubricating oils alone. If elasto-hydrodynamic regime is not reached, then the surfaces will be in direct contact with each other during operation, which is commonlly referred as the boundary lubrication (BL) regime. Under a boundary lubrication regime, the reduction of wear can be accomplished via surface-interaction, extreme pressure (EP) additives. Chemisorption of EP additives to various surfaces followed by formation of molecular boundary layer is the most common process, which can reduce wear behavior in materials. The future of better wear-reducing additives has been suggested to follow two main routes: (i) a traditional route focusing on molecular liquid additives which can interact with surfaces chemically to form protective films, or (ii) a non-traditional route involving solid, nanoparticle-based additives which can be present in the asperities regions to carry load and reduce friction and wear behavior [2]. Concurrent with the recent shift to more environment-friendly technologies, interest in the application of nanopowders (including ceramic) in lubricating oils has grown. However such particles are still not widely employed due to a general lack of understanding about their behavior under frictional pressure.

The molybdenum disulfide (MoS_2) based composite grease which is used in constant velocity (CV) joints in front wheel drive cars probably is the most common ceramic additive used today [3]. However, when MoS_2 is oxidized, it forms an abrasive molybdenum oxide MoO_3 that is undesirable. Among other materials, graphite also is frequently used [4], which displays low adhesion to surfaces and easily gets removed from interfaces. Moreover, lubricity of graphite also depends on adsorbed gases [5], which can result in performance variation depending on application environment. With the reduction of powder size to a smaller scale, there are many new materials that display improved lubricating properties, although there are not considered lubricants at the bulk scale or even in micron-scale. Such examples include nanodiamonds [6], carbon materials [7] or some nano-metals [8]. Several ceramic nano-powders have recently been tested as anti-wear and EP additives such as oxides [9], sulfides [10, 11], and nitrides [12]. A general characteristic that has been observed is that an optimal concentrations exist where below which those nano-powders improve wear behavior while higher doses have the reverse effect [12, 11].
In many cases, the cost of nanoparticle manufacturing is a limiting factor to their wider usage [13]. However, among the economically viable nanopowder additives, talc powder is commercially available as a filler for plastics. Talc ($Mg_3Si_4O_{10}(OH)_2$) is a 2:1 layered

magnesium hydrosillicate that is composed of an octahedral magnesium hydroxide layer sandwiched between two tetrahedral layers of silica. Those layers are weakly bonded together, giving talc its remarkable softness under sheer deformation. Talc is the softest known mineral with a hardness of 1 on the Mohs' scale. It is already widely used as a solid lubricant in medicine [14] and plastics [15] and extensive testing has been done on its safety [16]. Moreover, specific properties of talc such as hydrophobicity, and inertness are also well established and beneficial in wet environments where corrosion can be a problem [17]. Talc has been known for centuries as a solid lubricant and was studied in that role extensively, but rarely as a part of an oil-based lubricating composition [18, 19]. Due to its low environmental toxicity, talc can be used as a part of lubricating formulations with applications in total-lubrication-loss systems such as two-stroke engines and environmentally-conscience systems such as farming as well as hydropower. Serpentine ($Mg_3Si_4O_{10}(OH)_2$) is a 1:1 layered magnesium hydrosillicate that is composed of an octahedral magnesium hydroxide layer attached to tetrahedral layer of silica.

The goal of our study is to expand understanding of magnesium silicates in industrial lubricant applications to formulate suitable compositions. The main objective is to evaluate influence of concentration, temperature, and pressure on friction properties. We hypothesize that the interaction of talc and serpentine with a surface is different under room temperature versus elevated temperature and that the main mechanism of this change is powder dehydration. An understanding of any temperature dependance will allow the formulation of additives for specific applications. The main objective of this study is to evaluate the tribological properties of powders as an environmentally friendly ceramic extreme pressure additive to lubricating oils.

2.0 Experimental procedure

2.1 Materials

A commercial mineral energy-conserving engine oil (API SM grade 5W-30, O'Reilly, USA) was used as a base oil without further processing. The typical parameters of the oil provided by the manufacturer are listed in Table 1. To avoid batch to batch variations, oil for every test came from the same container.

Parameter	Value
Specific Gravity, ($60^{\circ}F$)	0.865
Viscosity, @ $40^{\circ}C$, cSt	52.0-71
Viscosity, @ $100^{\circ}C$,	9.3-12
Viscosity Index, Min	150
Flash Point, $^{\circ}C(^{\circ}F)$, Min	177(350)
Pour Point, $^{\circ}C(^{\circ}F)$	-29(20)
Color	2.0-4.5

Table 1: O'Reilly 5w30 oil product specification (provided by manufacturer)

To guarantee filter penetration in lubricating systems where the typical cutoff size is between 5 and 20 microns, we have to employ powders of the finest grind. One of the finest grinds of talc that is commercially available is Vantalc 6H-II (Vanderbilt, USA). Powder particle size distribution as determined by the manufacturer is as follows: LD20 200nm LD50 1.0μm LD90 2.5μm. Manufacturer provided chemical composition is represented in Table 2 and technical parameters in Table 3.

Chemical Analysis (calculated as oxides):	% by weight
Magnesium Oxide (MgO)	31.5
Silicon Dioxide (SiO_2) - by difference	61.4
Calcium Oxide (CaO)	0.2
Aluminum Oxide (Al_2O_3)	0.6
Ferric Oxide (Fe_2O_3)	1.1
Sodium Oxide (Na_2O)	<0.1
Loss on Ignition ($1000^{\circ}C$)	5.2

Table 2: Vantalc 6H-II chemical analysis data (provided by manufacturer)

Parameter	Value
Density at $25^{\circ}C$, mg/m^3	2.8
pH (ASTM D 1208)	9.4
G.E. Brightness, (TAPPI T 646)	>91
325 Mesh, %	trace
Oil Absorption (ASTM D 281)	55
Hegman Fineness (3 lbs/gallon)	6
Einlehner Abrasion Loss, g/m^2	8
Moisture, %	<0.5
Median Particle Size SediGraph 5100, μm	1.0
Bulk Density, $kg/m^3 (lbs/ft^3)$	240(15)

Table 3: Vantalc 6H-II typical technical properties (provided by manufacturer)

A Siemens D500 Kristalloflex X-ray diffractometer using copper K_{α} radiation at 30 kV and 30 mA at the room temperature was used to determine phases in the powder with a Ni-filter over the 2Θ angle range between 5° and 40°. A step size of 0.02° and a count time of 0.5 seconds were used for powder characterization. The powder was fixed on the observation glass with acetone and dried at the room temperature.

The surface area was measured using Brunauer, Emmett, and Teller (BET) surface area analysis (Tristar 3000, Micromeritics, Norcross, GA, USA). Before measuring the surface area, samples were dried in the sample holder at $200^{\circ}C$ for 2h, in presence of flowing N_2. The particle size distribution was measured using a dynamic light scattering (DLS) particle size analyzer NICOMP 380 DLS (Particle Sizing Systems, FL, USA) using an ultrasonicated solution of particles in deionized water. Samples of powder and wear tracks were observed under a field-emission scanning electron microscope (FESEM), equipped with an secondary electron Everhart-Thornley Detector (ETD), in high vacuum at 30 kV acceleration voltage with no applied coating (FEI Inc., Hillsboro, OR, USA).

2.2 Wear and friction test

A reciprocating ball-on-plate wear-testing was performed on the tribopair using a tribometer (NANOVEA, Microphotonics Inc., CA, USA) with 3 mm diameter hardened chrome steel ball (100Cr6, 58-63 HRC) rubbing against the flat test samples. 20 mm square wear test bottom samples were used. Before each test, all samples were cleaned with 99% isopropyl alcohol followed by 100% acetone to assure a clean surface as recommended by the ASTM133(2010) Section 3. In other parameters, we have used ASTM133 as a guideline for performing tests except the parameters below. Wear tests were performed under a normal load of 5*N* and at a sliding speed of 40*mm*/*s*. Lubricating oil was applied in a temperature regulated bath. The oil level was chosen to cover samples completely but prevent splashing. Each experiment was run for a distance of 2000 m. Friction coefficient was measured with sampling rate of a 100*ms*. The resulting file was digitally processed and the progression of static and dynamic coefficients of friction were extracted from the raw data. The static coefficient of friction was defined as the maximum coefficient of friction in the cycle and dynamic was calculated as the running average excluding maximums and minimums.

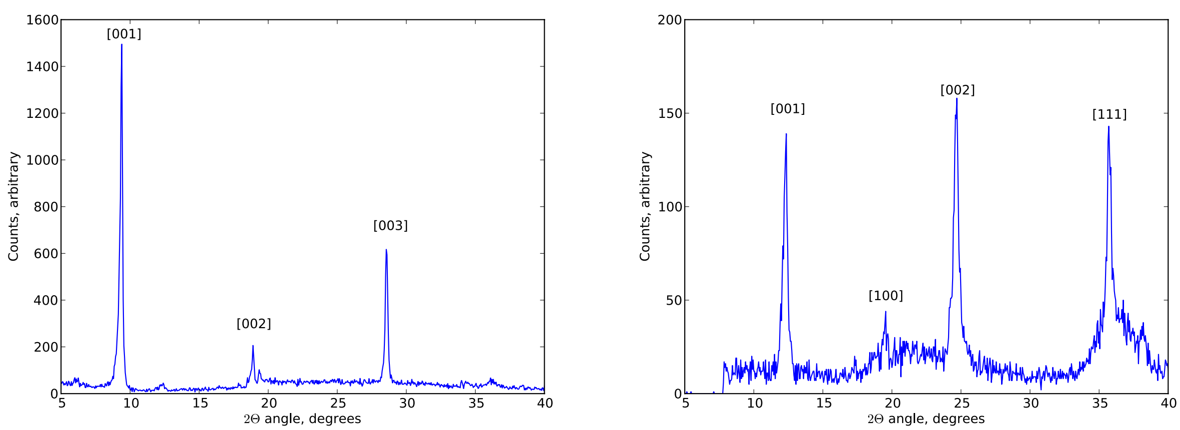

Figure 1. XRD spectra of nanopowders talc(left) and serpentine (right)

3.0 Results

3.1 Powders characterization

The XRD derived characteristic diffraction peaks at $2\Theta = 9.362^{\circ}$, 28.561°, 18.882° correspond to planes [001], [003], [002] spacing of 9.4391Å, 3.1228Å, 4.6961Å, respectively. The above peaks are identified as talc (JCPDS card No. 19-770). The XRD morphology index, as defined by Murtagh et al. [22] for talc was calculated based on the [004] and [020] plane peaks to be 0.614, which correspond to elongated plates. A BET surface area of 119.65±0.35 m^2/g was measured. XRD spectrum is presented in Figure **Error! Reference source not found.**. Dynamic light scattering is used to measure particle size distribution and found that LD50 is 0.9μ*m,* which agrees with the manufacturer provided data.

3.2 Powder dispersion in oil

To achieve consistent results, it was important to have a good dispersion of powders in oil. Initially different coupling agents were used as a dispersants. All coupling agents were selected on the basis of having the following characteristics: good solubility in oils, Ph above 7 and chemical stability at elevated temperatures. As has been demonstrated before [23], widely used silane agents are not capable of dispersing magnesium hydrosilicate powders in oil. Instead, we used *Ti*- and *Zr*- based surfactants (Ken-React Petrochemicals, NJ, USA). Five different commercial coupling agents: KR-TTS, LICA12, LICA38, ZR01 and ZR12; were tested with two transfer fluids: acetone and isopropyl alcohol. In all cases, the coupling agent was first dissolved in transfer fluid at a concentration of 1wt.%. Then, transfer fluid was mixed with powder in equal amounts by weight and the transfer fluid was evaporated by drying at $80^{\circ}C$ in an oven. The resulting powder was mixed ultrasonically with lubricating oil for 12h at $60^{\circ}C$ in a glass beaker using an ultrasonic mixer (Bransonic 220, Branson Ultrasonics Corporation, CT, USA). The stability of the dispersion was evaluated by sedimentation for a week at room temperature. The adequate dispersion and stability of powder was achieved using LICA38 with isopropyl transfer liquid. Based on these results, for all other tests we have used LICA38 as a coupling agent. Notably Zirconium based coupling agents did not offer adequate dispersion stability with our surface modification technique.

3.3 Effect of oil temperature

To evaluate the friction coefficient behavior as a function of temperature, we ran experiments with both powders at different temperatures. The progression of the friction coefficient, μ, as a function of time for talc is presented in Figure **Error! Reference source not found.**. Clearly there are two distinct phases of test: (i) the initial, intensive run-in stage that is characterized by substantially higher dynamic (0.25-0.3) and static (0.15-0.18) coefficients of friction with sudden changes, and (ii) the later steady state stage when coefficients of friction are essentially stable and substantially lower. The lowest steady state friction coefficients observed in the final stage were achieved at temperatures between $80^{\circ}C$ and $100^{\circ}C$. This particular temperature range is typically used in internal combustion engines and gearboxes, providing wide potential application area where these compositions can be potentially used. In the comparison to talc preliminary results of serpentine powder demonstrate effect at lower even room temperature when talc powders was less efficient.

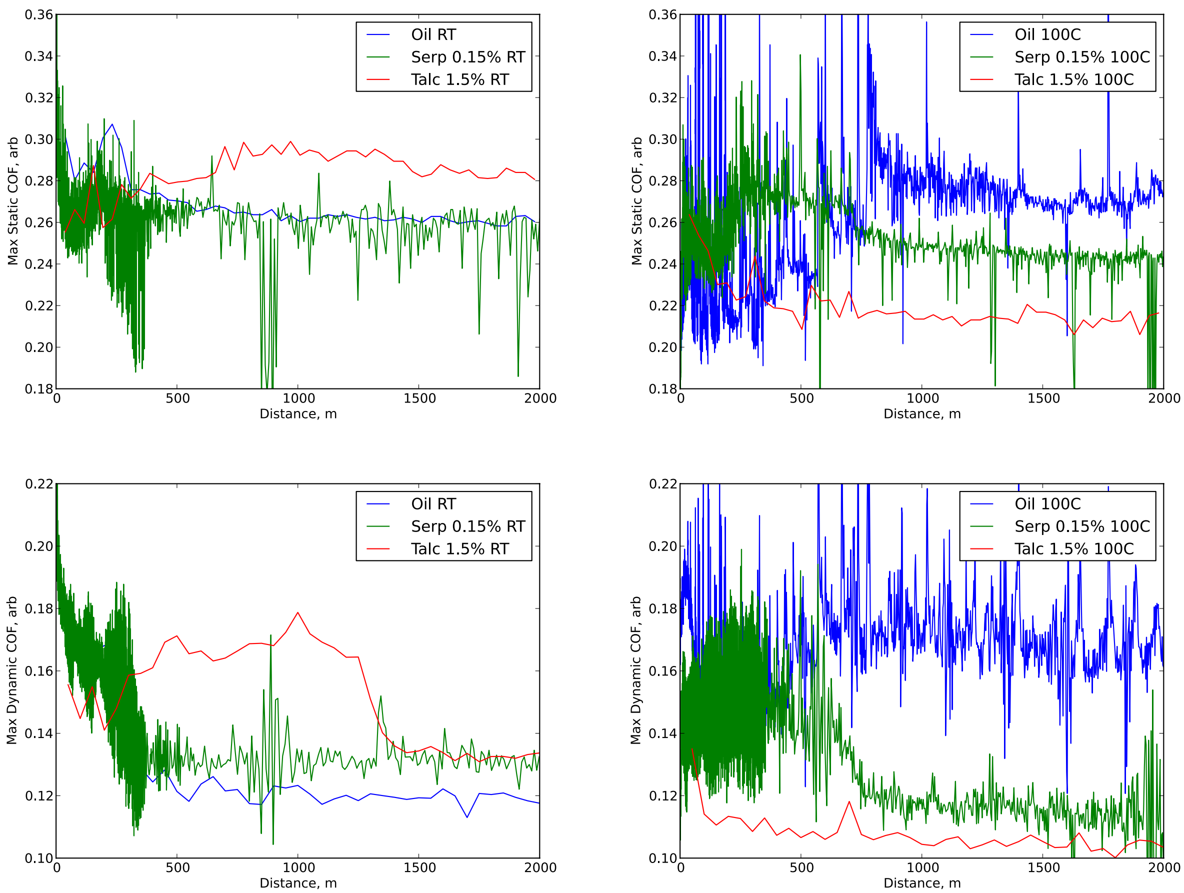

Figure 2 Effect of temperature on static (top) and dynamic (bottom) friction coefficient by talc and serpentine nanopowder additive to lubricating oil at room temperatures (left) and 100C(right). At the room temperature serpentine is more efficient but at 100C talc is more efficient than serpentine.

3.4 Effect of powders concentration

As has been noted before, the optimal concentrations vary with different powder materials [4]. This variation can be attributed to different particle size and morphology [25, 26] as well as variation in their surface reactivity [27]. To evaluate the optimal concentration of talc powder as a friction and wear reducing additive, several experiments were conducted at various concentrations. The working temperature was chosen at $100^{\circ}C$ for optimization. Time propagation of dynamic and static friction coefficients for various concentrations are displayed in Figure 3. After the initial run in, the friction coefficient stabilized and optimal results were achieved with a 0.15% concentration of talc in oil. In this best case scenario the dynamic and static friction coefficients were 0.15 and 0.11, respectively, which is more than 30% lower than 0.28 and 0.17 for pure oil under the same experimental conditions.

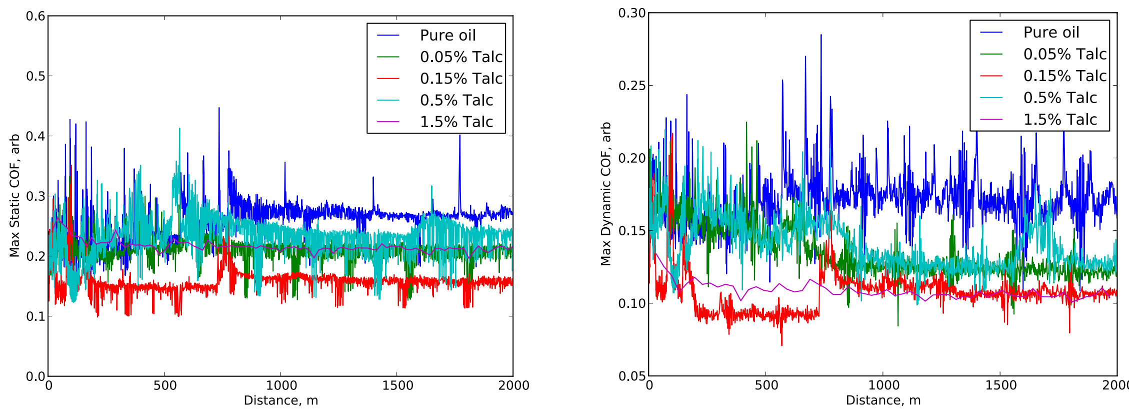

Figure 3 Influence of MHS powder concentrations in 5W30 oil on the friction coefficient at 100°C. The optimal concentration is determined as 0.15% for talc.

3.5 Microstructural observations of worn surfaces

Figure 4 shows a low-resolution SEM image of wear scars lubricated with pure oil and with talc powder additive. The pure oil image clearly displays an abrasive character of wear with long, sharp-edged grooves along the track. In contrast, any amount of added powder demonstrated remarkable smoothness of the surface. High resolution images confirm the abrasive wear in the case of pure oil and demonstrate the dielectric transfer film that can be identified by the charging artifacts on the edges. The micro-roughness of the wear tracks is greatly reduced with addition of nanopowders. The formation of tribofilm can be observed at the edge of the track and in more detail at higher magnification. The transition from the impact wear mechanism on the end of the scar to abrasive wear can be readily observed, in which abrasive wear is less prominent in the presence of talc powder. The detailed description of a film formation will be reported elsewhere.

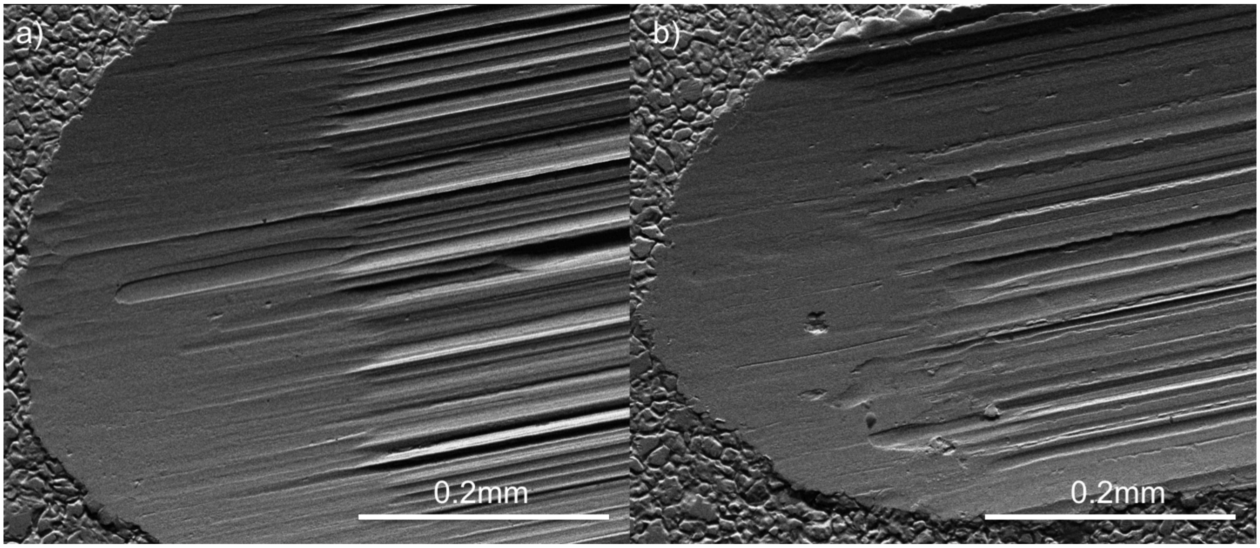

Figure 4 Wear scars of pure oil (a) and oil with 1.5% talc nanopowder (b) Abrasive wear is prevented by addition of nanopowders.

4.0 Conclusions

Magnesium hydrosilicate powders such as talc and serpentine in the nano to micron size range was evaluated as a friction-reducing additive to lubricating oil under various temperatures and concentrations. The efficiency in reduction of friction and wear has been shown in broad range of concentrations fo both materials. The final friction coefficient was reduced by more than 30% at the temperature 100C, but increased at room temperature for talc. Serpentine is more efficient at reducing friction at room temperature but less efficient at high temperatures. Pressure- and temperature- induced mechanochemical film formation was observed. The efficient dispersion of talc and serpentine powders in mineral oil can be achieved with LICA38 titanate coupling agent. Our results indicate that talc and serpentine nanopowders can be successfully used as an environmentally friendly extreme pressure (EP) and antiwear additive to formulate hybrid, liquid-based oil compositions.

5.0 Acknowledgement

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