Development and manufacturing an automated lubrication machine test for nano grease

Alaa Mohamed a,b,*, Shady Ali b, T.A. Osman c, Bahaa M. Kamel d

a Egypt Nanotechnology Center, EGNC, Cairo University, 12613 Giza, Egypt
b Department of Mechatronics, Canadian International College, Fifth Settlement, New Cairo, Egypt
c Mechanical Design and Production Engineering Department, Cairo University, 12613 Giza, Egypt
d Mechanical Engineering Department, National Research Centre, 12622 Giza, Egypt

Abstract

The present work deals with the development of nanocomposite grease to reduce friction and wear between two contact surfaces such as the components of engine, cams, gears, and bearing. The tribological and rheological characteristics of nano grease with a new design of automated lubricant machine tests were evaluated. In this paper, a manual Reicher test machine is converted to an automatic machine by an electric actuator, which used to convert the load from manual to automatic. Calcium grease containing a different concentration of titanium dioxide and carbon nanotubes TiO2/CNTs (0.5, 1, 2, 3, 4 wt. %) was successfully prepared. The tribological and rheological characteristics of the nano grease were investigated under different temperatures. The morphologies and chemical composition of the worn surface and the wear mechanisms were also discussed at different loads. X-ray diffraction (XRD) and scanning electron microscope (SEM) analysis were used to analyze the microstructure of the nano additives and nano grease. The result shows that the optimal concentration of TiO2/CNTs was 3 wt%, which reduces the wear and coefficient of friction about 72.3 and 60 %, respectively. Furthermore, the apparent viscosity and shear stress are increased by 48.2 and 74.2 %, respectively.

© 2019 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

Lubrication is one of the useful technologies, which acts as a layer of separation avoiding asperities of the surfaces getting into contact. Friction and wear are considered two major reasons for the failure of vital engineering components in various systems such as gears, pin joints, pistons, bearings, camshafts, pumps, compressors, and turbines [1,2]. To overcome this problem, lubricants are used in the engineering system. Lubricants help to minimize friction and wear in contacting surface in order to reduce friction and minimize power loss. In certain situation, lubricants fail to satisfy their lubricating properties. In order to improve the attributes of lubricants, nanotechnology offers the opportunity for improving the performance of lubricant oil using nano additives [3,4]. The addition of nanoparticles to base oil will enhance the certain characteristic of lubricant oil, such as improving friction, wear resistance, load carrying capacity, thermal properties, and rheological properties [5,6]. Therefore, attention has been paid by many engineering designers to investigate the wear...
behavior of materials under different operating parameters such as applied load, sliding distance, velocity, [7,8], and the contact conditions mechanism such as point, line, and area [9–12]. Furthermore, rheological characteristics such as yield stress, shear thinning, dropping point, consistency and thixotropic behaviors are strongly related to structure. Rheological properties of grease and structure are two important parameters which define “grease properties”. Therefore, few studies have been reported in the literature to improve the rheological properties by adding an additive like nanoparticles and thus to improve the greasing properties [13–15]. Moreover, the thermal conductivity of lubricant plays an important role in the transfer of heat during the contact parts in the lubrication process [16,17]. Therefore, suspending nano additives such as graphene or carbon nanotubes, which possess high thermal conductivity increases the heat transfer in grease due to increased thermal conductivity [18]. Based on the composition of nano additives and fluid, the thermal conductivity of nano grease may increase or decrease [19,20].

Oil analysis, is the laboratory analysis to determine a lubricant’s characteristics, pendent contaminants, and wears debris. This analysis helps in forecasting the life of a particular machine and hence the trends can be established, which intern help eliminate costly repairs. In the past few years, there has been a major development in the mechanical field in general and the mechatronics field in particular to create the simplest way to control any testing mechanism without human interference [21,22]. In this research, we are going to discuss control mechanism evolution from manual test to automatic test, which will reduce the errors that may occur from human interference.

Tribology is a very important language of mechanical engineers, covering lubrication sciences, friction, and wear. Many tribological apparatus have been designed and standardized to study the wear behavior of materials by the development of nanocomposite greases technology and deepening the understanding of the particularity of functional nanomaterials and nanoparticles. Nanoparticles used as additives show unique thermal, physical and chemical properties and have a broad application prospect in lubrication. Nano additives for lubricant such graphene nanosheets(GNS) [23,24], carbon nanotubes (CNT) [25–27], TiO2 [28–30], SiO2 [31,32], Al2O3 [33,34], ZnS [35] and MoS2 [36–39] influence distinguished anti-wear properties, dropping point, extreme pressure and good lubricating performances. Many researchers investigated the effect of GNS on the lubricating performance and the results cleared that the friction coefficient, the wear scar diameter, the extreme-pressure (EP) properties improved with 3 wt. % GNS. Additionally, the rheological properties of grease with different concentrations of GNS cleared that non-Newtonian behavior and the dropping point, apparent viscosity and shear stress improved by 17, 52, and 65 % respectively, with increasing GNS nanoparticles [23,24]. Bahaa et al. [40,41] studied the tribological and rheological characteristics of calcium grease containing carbon nanotubes. They reported that the optimum concentration of the CNTs in the nanocomposite was about 3 wt. % and the friction coefficients, wear scar diameter (WSD), shear stress and apparent viscosity were significantly improved with the addition of nanoparticles. Hui Wu et al. [28] studied the influence of TiO2 on the tribological properties of water-based lubricant and the results show that the optimal percentage of TiO2 was 0.8 wt. %, which demonstrates excellent tribological properties.

CNTs have attracted much interest due to their remarkable mechanical, thermal, electrical, chemical and optical properties [19,42]. CNTs are able to mitigate friction and wear in loaded contacts due to their shape, high aspect ratio, and high flexibility. In addition, previous studies reported that TiO2 has good friction-reducing, anti-wear, and cooling properties, so it can reduce the coefficient of friction and wear [43,44]. Therefore, recently researchers focused more on using a combination of two different nanoparticles in base oil or grease, which is state-of-the-art technology [45–47]. However, no investigation has been conducted yet on the combination of CNTs and TiO2 on the Tribological and rheological properties [48–50]. It is well known that nanocomposites usually show superior characteristics compared to the characteristics of each individual component due to the synergetic effects of more than one type of nanoparticles [51–53].

The aim of the present work is to update a new version of automated lubricant machine test, which converting control mechanism from manual test to automatic test, that will reduce the errors that may occur from the human interference. The hybrid nano additives TiO2 /CNTs reinforcing a calcium grease with different concentrations (0.5, 1, 2, 3, 4 wt. %) were investigated. Besides, the microstructure of these nano grease, the coefficient of friction, wear scar diameter, flow properties, consistency and dropping point of newly formulated greases are investigated.

## 2. Experimental

### 2.1. Materials

Commercial calcium grease (MERKAN 23) was purchased from Total Company. The physical properties of calcium grease are 96 mm2/s of viscosity at 40 °C, the dropping point is less than 100 °C and the penetration test is 245 mm at room temperature. Multiwall carbon nanotubes (MWCNTs) was synthesized by chemical vapor deposition with 99.9 % purity and the procedure is described elsewhere [54]. The physical properties of MWCNTs are 10–12 nm in diameter, 1–20 μm in length. Titanium dioxide powder (TiO2) (P25, 80 % anatase, 20 % rutile), was purchased from Sigma Aldrich. All chemicals used in these experiments were directly used without further purification.

### 2.2. Preparation of calcium grease containing TiO2 /CNTs

The preparation of TiO2/CNTs added in calcium grease is described as follows. Six samples, each of 150 g of grease were prepared with a mass fraction of TiO2/CNTs wt. % as 0, 0.5, 1, 2, 3, and 4 % respectively. First CNTs suspended in N,Ndimethylformamide (DMF) by ultrasonic probe were agitated for 30 min at room temperature in order to avoid the agglomeration of CNTs. In parallel, calcium grease was dissolved in chloroform (10 wt.%), at 25 °C for 1 h. After that, TiO2 and MWCNTs were mixed well in calcium grease by a high
homogenizer (SCILOGEX D500, USA) and magnetic stirrer for 30 min at room temperature to form a homogenous composite, which has the ratios of MWCNTs to TiO2 in the proportion of 1:1.

2.3. Characterization of TiO2/CNTs nano grease

The structural features of nanoparticles and nano grease were analysed by using high resolution transmission electron microscope (TEM, JEOL, JEM 1200) with an accelerating voltage of 20 KV. The samples were prepared by placing a small amount of nano grease on a sample grid and immersed for 10 min in hexane to remove the base oil. Then, left for drying for 15 min in an oven at 30–40 °C. Powder X-ray diffraction (XRD-RigaKu D/max- RB, Japan) using Cu Ka radiation (k = 1.54056 Å, 40 kV, 30 mA) was used to identify the characterization and the phase of MWCNTs and TiO2. The topography of worn surfaces of the lower steel balls was investigated by a JSM-5600LV scanning electron microscope (SEM-JEOL/JSM-5600LV) equipped with energy dispersive X-ray (EDX) to identify the morphology and the composition of the rubbed surface.

2.4. Determination of rheological parameters of nano grease

Rheological characteristics of the grease are defined as the deformation of a body under the effect of stress [55]. Therefore, these properties may be of importance in anaerobic digestion for the dimensioning. Rheological characteristics are measured by the shear stress (τ) related to shear rate (γ) [56], in which the shear stress is defined as the force (F) divided by the area (A) (Eq. 1). On another side, the shear rate (γ; s⁻¹) describes the velocity gradient (Eq. 2). A Rheometer (DV-II Ultra) was employed to measure the rheological behavior (shear stress) and viscosity at given shear rates of different samples of nano grease at the different temperature.

\[ τ = \frac{F}{A} = \frac{N}{m^2} = Pa \]  
\[ γ = \frac{dvx}{dy} = \frac{(m/s)}{m} \]

2.5. Determination of dropping point of nano grease

Dropping point is defined as the temperature at which, either the first drop of material falls from the cup or the grease passes from a semi-solid to a liquid state. The principle operation of the dropping point is a sample of the lubricating nano grease contained in a cup suspended in a test tube heated in oil. In addition, the temperature of the grease, at which the first drop falls from the cup, is the dropping point of the grease, given as per ASTM standards D-566 [57].

2.6. Determination of thermal conductivity of nano grease

Thermal properties are among the most important properties to investigate the thermo-physical properties of the nano grease. The KD2 thermal properties analyzer (1, 4, 24 and 25) was used to measure the thermal conductivity and thermal resistivity of the fabricated nano grease. The operation principle of the KD2 thermal properties analyzer is based on the transient hot-wires method [21]. The experimental setup, testing, and temperature control have also been reported in previous articles [46].

2.7. Friction and wear test of prepared greases

Different tribo-machines have been used such as a block on the disc (Fig. 1a), Block-on-Ring (Fig. 1b), four balls, Dry Sand Rubber Wheel (Fig. 1c), sand/steel wheel test under wet/dry conditions and so on to determine the tribological behavior. The basic differences between these techniques are the contact mechanism (area, line or point contact) of the tested material. The testing machine basically consists of a rigidly-mounted test roll, which is pressed against a revolving friction wheel by means of leverage. The friction wheel is immersed with its lower third in the sample under test. Its rotating speed is at a rate that ensures that a sufficient quantity of lubricant will always get to the contact surface of the test roll and the friction wheel. Due to the crosswise arrangement of the axles, an elliptical wearing surface will be formed, the size of which will grow corresponding to the operating time elapsed. The extension of the wearing surface results in a decrease of the specific surface pressure between friction wheel and test roll, whereby a load-carrying lubricant film will be built-up. Additionally, in this machine, the automated load is used instead of the manual load by setting an electric actuator that will move linear downwards and the test piece is attached at the end of the actuator, which will touch the oil that is rolling on the friction disc until it breaks.

\[ \text{Fig. 1 – Schematic drawing showing the most common configurations of tribological machine (a) block on disc, (b) block on ring and (c) dry sand rubber wheel [58].} \]
An electric linear actuator is powered by a motor to convert electrical energy into torque. An electric motor mechanically connected turns a lead screw (a threaded lead or ball nut with corresponding threads) that match those of the screw is prevented from rotating with the screw. When the screw rotates, the nut gets driven along the threads. Then, the direction of the nut moves depends on which direction the screw rotates and also returns the actuator to its original position. Moreover, the electric actuator was used for several sizes, loads and offers accurate speed and force controls. All the data were collected by a computer with a data acquisition system. The current design converts the block on ring machine from manual to automatic by using the electric actuator which the modified machine is shown in Fig. 2.

3. Result and discussion

3.1. Structural characterization of nano grease

The high magnification TEM images in Fig. 3 show the morphology of CNTs, TiO\(_2\), base calcium grease, and modified calcium grease with nanoparticles. Fig. 3 (d) shows that there is no apparent aggregation of TiO\(_2\)/CNTs in grease. It is also observed that the structure of the modified calcium grease is regular and homogeneous network structure, which confirms the rheological and tribological stability as reported in previous articles [59].

3.2. Tribological characteristics of the nano grease

Tribological characterization of the modified calcium grease was carried out to evaluate the wear scar diameter (WSD) and the coefficient of friction (COF) at different concentrations (0.5, 1, 2, 3, 4 wt\%) of TiO\(_2\) / CNTs. It was found that the optimum concentration of the TiO\(_2\)/CNTs is 3 wt\% where the WSD was reduced by 66 \%, then it started to increase as shown in Fig. 4. The nanoparticles form a tribo-film at the interface, which acts as a layer of separation to avoid asperities of the surfaces getting in contact and helps in reducing the wear of the specimen [60]. Fig. 5 shows the effect of the friction coefficient at different concentrations of TiO\(_2\)/CNTs NPs. The friction coefficient of calcium grease having TiO\(_2\)/CNTs decreases gradually with increasing TiO\(_2\)/CNTs within the range of 0.5–3 wt\% because NPs act as a load bearing element and reduces the friction between the surfaces. Then, increased with increasing the concentration of TiO\(_2\)/CNTs NPs to 4 wt\%. This is mainly caused by the agglomeration of the nanoparticles.

3.3. Tribomechanical mechanism of nano grease

Fig. 6 shows the morphologies of the wear scar lubricated by calcium grease and by 3 wt\% of TiO\(_2\)/CNTs calcium grease at 150 N for 60 min. The scuffing and scratches on the worn surface lubricated by 3 wt\% of TiO\(_2\)/CNTs were smaller than those lubricated with the base grease. These indicate that a certain percentage of TiO\(_2\)/CNTs additives in calcium grease improved the anti-wear effects in the tribological test.

3.4. Rheological behavior of characteristics of the nano grease

Fig. 7 shows the shear rate as a function of shear stress for different concentrations of TiO\(_2\)/CNTs (0.5, 1, 2, 3, and 4 wt\%) at room temperature. The results presented that the shear stress was nonlinear with the shear rate for all the different concentrations, which indicates that the grease has a non-Newtonian behavior [61]. It’s evident that the slope of the line increases with increasing different concentration of TiO\(_2\)/CNTs from 0.5 to 4 wt\%, that is observed in many articles [62,63], but the improving at concentration 4 wt\% is unsatisfactory. This is mainly caused by the agglomeration of the nanoparticles.

The effects of apparent viscosity versus shear rate for base grease and base grease with different concentrations of TiO\(_2\)/CNTs NPs are shown in Fig. 8. The viscosity of nano grease becomes larger with the decrease of shear rate and increases with increasing of the concentration of TiO\(_2\)/CNTs as an additive on calcium grease. The shear stress and apparent viscosity of the calcium grease having 3 wt\% of TiO\(_2\)/CNTs are much higher than that of pure calcium grease at all shear rates, which the apparent viscosity and shear stress are increased by 48.2 and 74.2 \%, respectively.
Fig. 3 – TEM image of (a) Multiwall carbon nanotubes, (b) Titanium Dioxide, (c) Base calcium grease, and (d) nano grease.

Fig. 4 – Wear scar diameter versus different TiO$_2$/CNTs concentration.

Fig. 5 – Friction coefficient versus different concentration of TiO$_2$ / CNTs.

Fig. 9 revealed the influence and relationship between the apparent viscosity of nano grease and the shear rate at different temperatures 25, 75 and 100 °C. A shear-thinning (pseudo plastic) behavior was observed. On the other hand, it is clear from Fig. 9 that the temperature has a strong effect on the rheological characteristics of nano grease to be a non-Newtonian fluid at high shear rates. The results indicate that the viscosity was dependent on the shear rate (Shear Thinning or Thickening) or the deformation history (Thixotropic fluids) and Newtonian fluid at low shear rates. Besides, the results are in agreement with many previous studies [40,51]. Actually, the viscosity of nano grease reduces with the increase of temperature at a different concentration of TiO$_2$/CNTs, which is reported for a wide class of nano grease [64,65]. Furthermore, at low shear rates, the apparent viscosity of nano grease increases with increasing temperature, showing a stronger shear-thinning behavior and at 25 °C is stronger than at 75 and 100 °C.
3.5. Effect of TiO$_2$/CNTs additives in dropping point

Dropping point as a function of different concentrations of TiO$_2$/CNTs with calcium grease was presented in Fig. 10. The result shows that the dropping point of nano grease is extremely improved by adding TiO$_2$/CNTs gradually from 0.5 to 4 wt%. This result shows that the TiO$_2$/CNTs as an additive was effective in enhancing the dropping point of base grease which increases about 31.9%.

3.6. Effect of TiO$_2$ CNTs additives on thermal conductivity of nano grease

Fig. 11 presented the effect of TiO$_2$/CNTs on the thermal conductivity of base grease at different concentrations. The result indicates that the thermal conductivity of nano grease is increased significantly linearly with increasing the additives, which show that the TiO$_2$/CNTs as an additive play significant role in the thermal conductivity improvement of nano grease, which increases about 53.4%. The main cause of improving the thermal conductivity is that at a higher volume concentration, the number of suspended nanoparticles is higher. Therefore, this leads to the enhancement of the ratio of surface to volume and collisions between particles [66,67].

Fig. 6 – SEM morphologies of wear scar a) base grease, and b) nano grease with 3 % wt. of TiO$_2$/CNTs at 150 N for 60 Min.

Fig. 7 – Shear stress versus Shear rate for base grease and grease with different concentration of TiO$_2$/CNTs.

Fig. 8 – Apparent viscosity versus shear rate for base grease and grease with different concentrations of TiO$_2$/CNTs.

Fig. 9 – Temperature dependence of the shear thinning (non-Newtonian) viscosity of nano grease.
increasing the TiO$_2$/CNTs, which increased about 53.4 %. This enhancement is attributed to the nanoparticles that form a tribo-film on the contact surfaces.

**Conflict of interest**

The authors declare that they have no conflict of interest.

**References**


nanofibers/biogenic silica composite nanofibers. Sep Purif Technol 2019;210:935–42.


