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Influence Of Nano Grease Composite On Rheological Behaviour

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Abstract

The aim of this work is to study the rheological behaviors of carbon nanotubes (CNTs) as an additive on lithium grease at different concentrations. The results indicated that the optimum concentrations of the CNTs was 2 %. These experimental investigations were evaluated with a HAAKE Rheovisco RV20, Penetrometer and Measurement of the dropping point. The results indicated that the shear stress and apparent viscosity increase with the increase of CNTs concentration, penetration and consistency not effect of base grease, and the dropping point increasing about 25%. The microstructure of CNTs and lithium grease was examined by high resolution transmission electron microscope (HRTEM) and transmission electron microscope (TEM).

***Keywords*:** Carbon nanotubes, Rheological behavior, Lithium grease, Microstructure.

I. INTRODUCTION

Grease is a solid or semi fluid which would normally have been employed together with a thickener, additive and anti-oxidant agent. The fluid lubricant that performs the actual lubrication can be petroleum (mineral oil), synthetic oil, or vegetable oil. The thickener gives grease it characteristic consistency and is sometimes believed as a “sponge” that holds the oil in place [1]. The majority of greases on the market are composed of mineral oil blended with a soap thickener. Additives enhance the performance and protect the grease and lubricate surfaces. The influence of the rheological properties of CNTs additives is very important for all the grease lubricating bearings. To characterize a lubricant comprehensively, the rheological properties at all working conditions, pressures and temperatures have to be known [2, 3]. Grease is widely used as a lubricant in the wheel assembly, journal bearings and rolling element bearings. Grease is also used in other areas that need occasional service like the brake or stopper assembly to help keep these fittings rust free and make removal of dirt and grime easier. Grease is applied to machines that can be lubricated infrequently and where lubricating oil would not stay in position. It also act as a barrier to prevent entering of water and the incompressible materials. CNTs used as a performance enhancing additive in gear lubricants for extended lifetimes, lower operating costs, and improved power efficiency. Numerous laboratory investigations and industrial experience indicate that using of CNTs has significant advantages compared to conventional solid lubricants in both mild and extreme pressure conditions [4-6].

Lubricating grease consistency has been evaluated for years with cone penetration test ASTM-D217. The test measures the distance in tenths of a millimeter to which a standard metal cone will penetrate into the grease surface under standard conditions. This single numerical value has been proven to be inadequate to estimate the real consistency of lubricating grease under dynamic conditions. It ignores the non-Newtonian flow behavior characteristic to grease. In the past few years, rheology has been introduced as a new method to better understand and evaluate the real behavior of lubricating grease. Rheology takes into account the influence of shear rate, shear stress, temperature and time. By measuring the viscosity with both rotational and capillary rheometer, it is possible to see the effect of shear rate on grease consistency which strongly influences the lubricating capability of greases under load [7, 8].

The aim of this work is to evaluate the rheological behaviors of carbon nanotubes (CNTs) as an additive on lithium grease at different concentrations and study the microstructure of lithium grease.

II. EXPERIMENTAL METHODOLOGY

2.1. Syntheses of Carbon Nanotubes and Lithium Grease

CNTs were synthesized by the electric arc discharge. The arc is generated between two electrodes (size φ 6 x 100 mm) using distilled water. The cathode and the anode are from graphite (99.9% pure), and was performed under AC current, 75 A and 238 V.

Grease that was used in this work was commercially available; the main physical-chemical properties of the grease are presented in Table 1. The grease is lithium based and has good heat-resistance, water resistance and mechanical stability. In order to study the rheological behavior of carbon nanotubes as an additives on lithium grease, carbon nanotubes were added into lithium grease at the different concentrations (0.5, 1, 2, and 3 wt. %). The carbon nanotube particles were dispersed well in the grease in an ultrasonic bath.

Table 1: Composition of the tested grease

Base oil	Mineral oil
Soap thickener	Lithium
Penetration (1/10 mm at 25°C)	280
Dropping point	180 °C
Viscosity of base oil at 40°C	150 cSt

2.2. Structural Characterization

The size and morphology of carbon nanotube were characterized with high resolution transmission electron microscopy (HRTEM) (JEOL JEM 2100) with an accelerating voltage of 200 kV.

The grease structure was investigated transmission electron microscopy (TEM) (JEOL JEM 2100) with an accelerating voltage of 200 kV.

Transmission Electron Microscopy (TEM) observations were conducted after a classical sample preparation. A small amount of grease was placed on a carbon coated sample grid and immersed for several minutes in hexane to remove its base oil. It was then dried for 15 minutes in an oven at 30° - 40°C.

2.3. Viscometer (HAAKE Rheovisco RV20)

This experimental investigation employed a commercial rotational viscometer, HAAKE Rheovisco RV20. The instrument consists of the base unit of Rotovisco RV20, the Rheocontroller RC20 which acts as an interface between the computer and Rotovisco RV20, and the measuring system M5 utilizing a cone and plate configuration. A HAAKE circulator provides precise temperature control for the samples. The operation principle of the instrument is illustrated in Fig 1.

After placing the grease sample in the gap between the cone and the stationary plate, the cone is driven to rotate at programmable speeds by a DC motor with a feed back loop for accurate speed control. The rotation of the cone leads to a uniform shear rate in the sample. The resistance of the sample to flow gives rise to a very small distortion in a torsion bar, mounted between the motor and the driven shaft. This distortion is detected by a transducer. Signals proportional to the speed and the torque are respectively transmitted to the control unit for processing. A flow curve plotted as shear stress vs. shear rate, which indicates the flow characteristics and is regarded as the rheological 'fingerprint' of the sample, is obtained. With a carefully designed test scheme, much more information about the sample's rheological properties can be collected.

Out of consideration for thixotropy, the test procedure should include a set holding time with the aim of degrading the thixotropic structure after measuring the flow curve from zero to a predetermined maximum shear rate, and then measuring a flow curve back to zero shear rate. If a hysteresis exists between the ascending and descending curves, the substance can be referred to as thixotropic and the area between the curves corresponds to the extent of thixotropy.

In the rheological measurements of grease with a cone and plate configuration there may be some anomalous phenomena such as slip at the wall, fracture and flow disturbance, which make the experimental results unreliable and should be avoided as much as possible.

Observations showed that slip at the wall begins to appear at shear rates lower than about 10 s^{-1} and becomes greater when the applied shear rate decreases. For a shear rate greater than 10 s^{-1}, the contribution of slip at the wall to the total strain becomes low compared with viscous deformation [9]. A characteristic of a measurement exhibiting slip at the wall is that the flow curve will shift if measured with a different sensor system geometry. It has been demonstrated to be a quick and efficient method for judging the presence of slip flow to measure the same sample under constant conditions with different sensor systems [10].

Fracture occurs systematically when the shear rate increases [9]. When a free surface forms in the grease film at the edge of the gap, the effective radius of sheared grease is reduced; as a result, the calculated shear stresses are erroneously low. The magnitude of the reduction in grease radius can be estimated from the size of the undisturbed annulus around the periphery of the grease film after withdrawing the cone from the plate [11]. It seems that when the angle and radius of the cone is small (i.e. the gap is narrow), the influence of fracture on the measurements is tolerable.

The flow disturbance is caused by the normal stress and inertia of the sample. It is negligible when the cone angle is small enough; the shear rate is not very high and the elasticity of the sample is not very significant [12].

In this investigation, the cone radius was 10 mm; the cone angle was 1°; another cone angle of 5° was employed for examining the validity of the test results. In addition, the tested grease was experimentally verified without significant elastic effect. The flow disturbance can therefore be neglected. Furthermore, the test was schemed and carried out with discretion. All the results were examined carefully to detect the influence of slip and fracture. Only those which were apparently not disturbed are presented here.

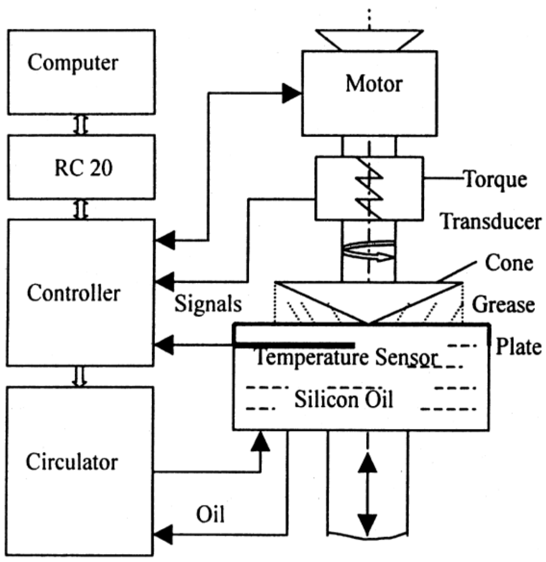


Fig. 1: The principle of HAAKE Rheovisco RV 20

2.4 Penetration and Consistency

The most important feature of grease is its stiffness or consistency. For oils the viscosity is measured to assess oil fluidity. For greases the penetration or the consistency indicates whether the grease is softer or more solid or stiff. Grease consistency depends on the type and amount of thickener used and the viscosity of its base oil. Grease's consistency is its resistance to deformation by an applied force. For use greases the consistency is measured by a Penetrometer as shown in fig. 2 with a quarter cone. The penetration is used as an identifier and provides information whether it can be pumped by a central lubrication system or used for a certain application [13].

2.4.1 Test Principle

2 g of the grease sample is filled at room temperature into a standard beaker. The tip of a standardized double cone touching the surface. Over a 5 second period how deep the cone penetrates into the grease is measured. Soft greases will have higher penetrations than hard greases.

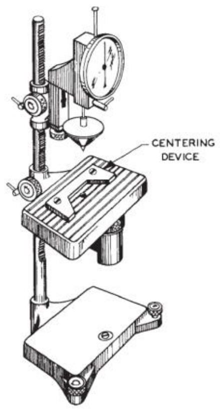


Fig. 2: Penetrometer

2.5 Dropping Point

The dropping point is the temperature at which the grease passes from a semisolid to a liquid state under the conditions of the test. The test shows the end point of a softening process under static conditions [14].

Dropping point indicates the upper temperature limit at which grease retains its structure, not the maximum temperature at which grease may be used. They are not thinned in a uniform way, they get softer dependent on the thickener type. For the determination of the operating temperature of the grease, the oxidation of the base oil and the destruction of the thickener but not the dropping point are more relevant (Fig. 3).

2.5.1 Test Principle

A small sample volume of approximately 0.5 g is filled into a nipple has an associated thermometer. The test unit is heated until a drop is formed on the bottom opening of the nipple. The drop, consisting of a thickener and oil will fall into the test tube. The temperature, at which the drop formation starts, is recorded as "dropping point". The test unit operates up to 300 °C.

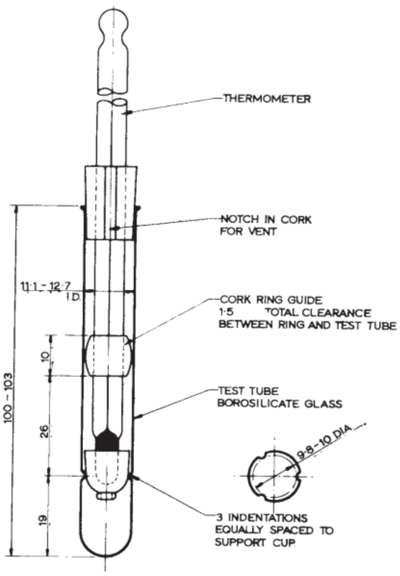


Fig. 3: Measurement of the dropping point

III. RESULTS AND DISCUSSION

3.1. Structural Characterization of Carbon Nanotubes

High resolution transmission electron microscope (HRTEM) image of CNTs shown in Fig. 4 show the presence of different structures in the sample and the average size of the nanoparticles is about 10 nm in diameter and 1-25 µm in length.

Figure 5 shows the SEM image of CNTs dispersed in lithium hydroxystearate (soap) fiber. It can be seen that there is no apparent aggregation of CNTs, indicating that the CNTs could be well dispersed in lithium grease, and it can be observed that the microscopic structure of lithium grease presents a more regular and homogeneous network structure, with long fibers, which confirm the rheological stability.

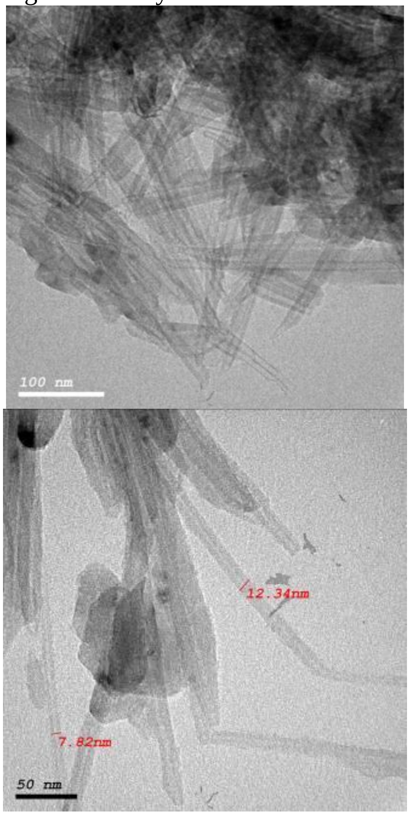
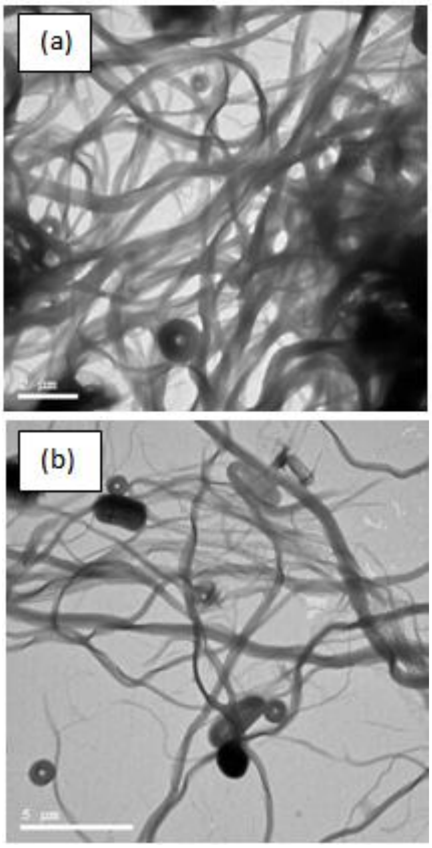


Fig. 4: HRTEM images of CNTs



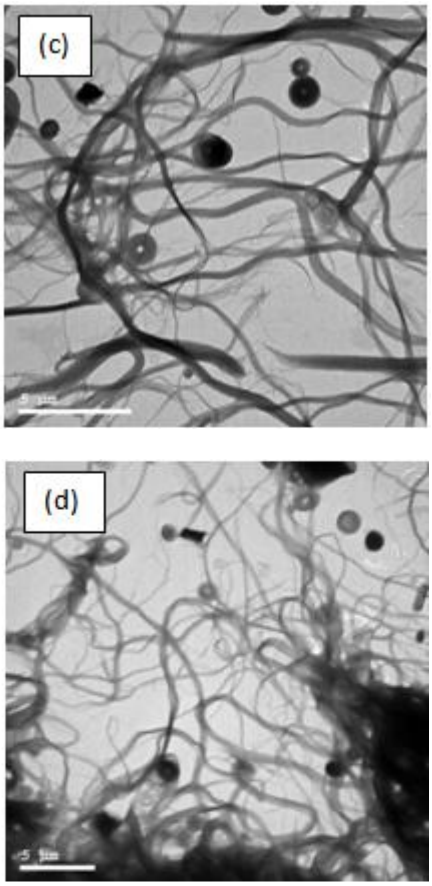


Fig. 5: TEM image of grease with (a) base grease (b) 0.5 % (c) 1 % (d) 2 %

3.2. Rheological Behavior of Carbon Nanotubes as an Additives on Lithium Grease

Many models are available to describe rheological properties of lithium grease such as; Bingham model, Herschel - Bulkley model, Casson model, Bauer model, Balan model, Papanastasiou model, Dorier and Tichy model.

The rheological results from the measurements with the cone and plate rheometer, that are shown in figures 6 and 7 represent the effect of carbon nanotube additives on lithium grease with shear stress and viscosity.

Figures 6 and 7 give the shear stress and apparent viscosity as a function of shear rate for lithium grease alone and that containing different concentrations (0.5, 1, 2, and 3 wt. %) of CNTs. It can be seen that the shear stress and apparent viscosity of the lithium grease containing 2 wt. % CNTs are much higher and more stable than that of pure lithium grease at all shear rates. At this point, the shear stress and apparent viscosity could be increased by 67.3 % and 81.8 %, respectively. The shear stress of base grease and the grease containing CNTs become larger with the increase of shear rate and with the increase of the percentage of carbon nanotube additives on lithium grease.

The apparent viscosity of base grease and the grease containing CNTs becomes larger with the decrease of shear rate and increases with increasing the percentage of carbon nanotube additives on lithium grease. These experiments were carried out under stationary conditions, to avoid thixotropic behavior. Therefore, the result indicates that all the samples show a large shear thinning behaviour. At low strain rates, the values of apparent viscosity follow quite well the classification found for the yield stress.

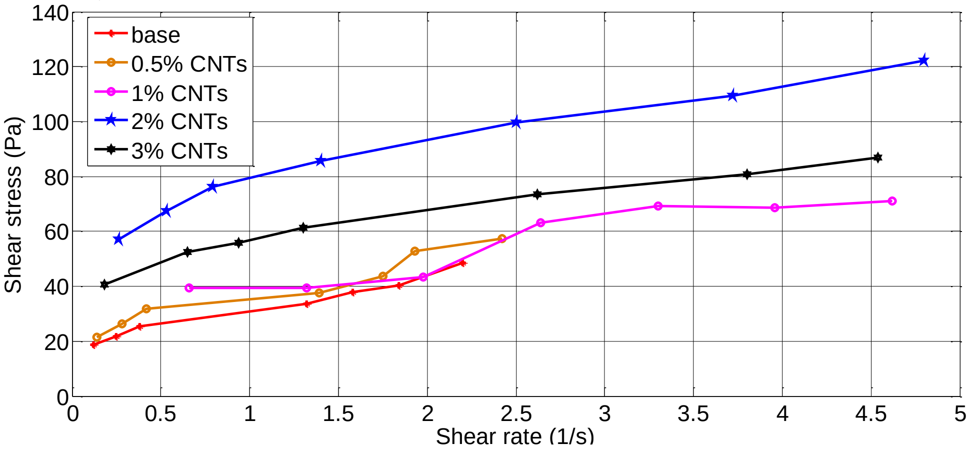


Fig. 6: Shear stress of the grease samples

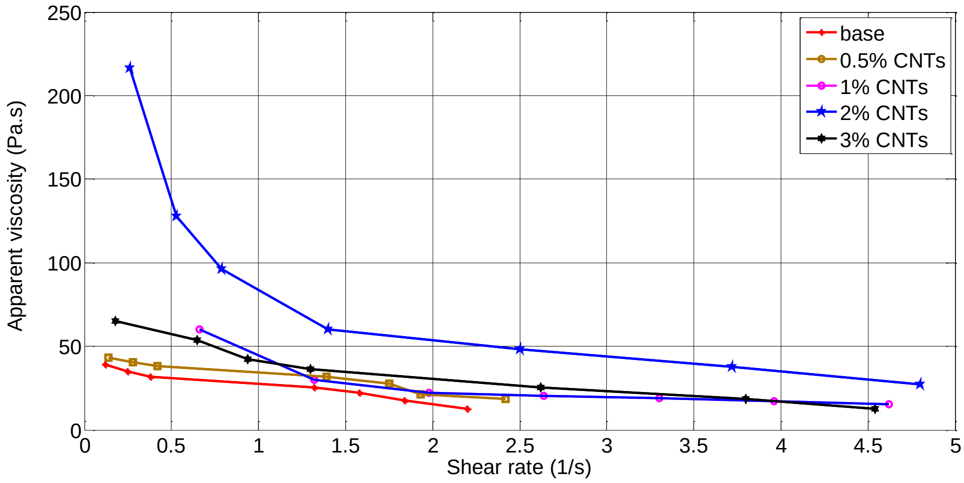


Fig. 7: Apparent viscosity of the grease samples

3.3 Penetration and Consistency

The consistency of the grease characterizes its ability to be deformed in an application. The consistency is grouped in NLGl classes from 000 to 6. If the used grease penetration is compared to the fresh grease, following information can be gathered:

1. The penetration will be higher if there is by water or other liquid contamination
2. The grease will be softer if it is sheered by mechanical stress in a bearing. This destroys the soap structure and shears its long fibered components.
3. The penetration is lower and the grease gets harder if it contains less base oil and more thickener. This may happen if base oil is lost by bleeding out because of vibrations or if it is vaporized by high temperature or oxidation.

Penetration and Consistency of CNTs added into lithium grease is the same of base grease, because thickener gives grease its characteristic consistency not additives. Therefore, the results indicating that the CNTs as an additive not an effect of base grease.

3.4 Dropping Point

The dropping point only indicates whether grease is running at a specific operating temperature. The maximum operating temperature for a grease should be always far below the dropping point temperature. The base oil type and the thickener will determine how far below the dropping point the operating temperature can be. Usually the dropping point should be at least 50 °C higher than the operating temperature.

Dropping point of CNTs added into lithium grease could be increased 25% at 2 wt. %. Therefore, the results indicating that the CNTs as an additive are effective in improving the dropping point of base grease.

IV. CONCLUSIONS

According to the above results and discussion, the conclusions can be summarized as:

1. CNTs were successfully synthesized by electric arc discharge method. The synthesized CNTs have an average diameter of 10 nm and could be well dispersed in lithium grease.
2. A rheological characterization, including apparent viscosity, shear stress and shear rate was carried out at different concentrations of CNTs. The grease response was studied at constant temperature and time, which led to a real mechanical spectroscopic investigation.
3. The microstructure of lithium grease at the different concentrations was confirmed by scanning electron microscope (SEM). The results indicated that the microscopic structure of the lithium grease presents a more regular and closer network structure with long fibers, which confirms the rheological stability.
4. CNTs as an additive are effective in improving the dropping point of base grease about 25%.
5. The optimum percentage of the CNTs in the grease composites was 2 %.

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