Investigation on Tribological Properties of Carbon Nano Tubes as Additive with Lithium and Calcium Grease

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Abstract - The multi-walled carbon nanotubes (CNTs) were added to lithium and calcium grease with varying percentage. CNTs with an average diameter of 10-15nm and an average length of 5μm was used and its structural characterization was carried out through electron microscopy. Tribological properties of grease added with varied percentage of CNTs were evaluated using a four ball tribometer. The obtained results revealed that, CNTs addition to grease reduced coefficient of friction and wear scar diameter (WSD) compared to neat base grease. The optimal values of results were obtained at 0.6wt.% addition of CNTs for better anti-wear and anti-friction characteristics of grease. Also, increased CNT to 6 weight % has resulted in smooth surface on contact area. The severely rubbed marks were evident on scanning electron microscopy (SEM) images of neat grease lubricated worn surface. Whereas, SEM images of CNT added grease lubricated worn surface revealed the scuff marks on contact area.

Keywords: CNTs; Lubricant; Tribology; Frictional Coefficient; Wear Scar Diameter;

I. INTRODUCTION

Industrial machine components such as bearings demand for better tribological characteristics viz. wear resistance, low friction and high speed lubricants. Grease is widely used type of lubricant and also as sealant in various engineering applications. It is generally available in the form solid and semi-solid. Commercially available grease typically comprises of polyalphaolefin based oil and metal-soap thickeners such as lithium, calcium and aluminum. The thickeners impart peusdo-plastic property to the grease. Besides, under static load condition, boundary film of lubricants collapses resulting in adhesion and surface damage to components. Thus, anti-wear additives were added to base grease to reduce surface damage caused with the application of load and generated friction [1]. In addition, different types of additives such as graphite, molybdenum disulfide, etc are blended to base grease. They play role as anti-wear additives, friction modifier, rust inhibitors extreme pressure stabilizers.Shafi and charoo[2] have shown that avocado oil blended zinc dialkyldithiophosphate improves the tribological performance of oil through the formation of stable tribofilm between pairs.

The addition of nanoparticles to lubricants has gained importance due to their contribution in enhancing lubricant tribological characteristics like reduction of friction, wear, etc[3]. Various researchers have employed metal nano-particles as additive to grease to achieve better lubrication performance characteristics[4–8]. Xue et al. [9] have shown that surface modified TiO_2 nanoparticles additive in liquid paraffin has enhanced its friction and wear characteristics. Zhou et al. [10] and Zhang et al. [11] have found that addition of Cu nanoparticles to lubricating oil reduces the friction and wear. Also, authors have observed the presence of Cu boundary tribofilm on worn surface. Liu et al. [7] have concluded that Sn and Al nano particle can improve antiwear and extreme pressure properties of lubricant. Wu et al. [12]reported that nano-cerium oxide (n-CeO_2) particles outperform as additive in improving tribological characteristics of poly-alpha olefin. The n-CeO_2 addition of 0.2% has resulted in better anti-wear property. Many researchers are paying attention to green additives. Perhaps environment concern lies in using nano-green additives over various other nanoparticles comprising heavy metals, sulphur or phosporous, etc. $CaCO_3$ nanoparticles are promising in this regard as an additive. Literatures [13,14] have reported that $CaCO_3$ nanoparticle addition has improved the friction and wear behavior through the formation of tribofilm containing $CaCO_3$ and CaO.

Over the last few years, carbon nano-tubes (CNTs) have attracted various researchers and engineers due to their unique physical and chemical properties in engineering applications [15,16]. In tribological applications, carbon nano-tubes were added with grease to reduce friction and wear [17]. Tonk[18] has presented state-of-art on CNTs as additives for lubrication. The study suggested that CNTs as lubricant additives helps in reducing friction and wear, thereby improves the performance of machinery and reduces frequency of maintenance. Also, forecasted that in another 5 years nanolubricants will be the incorporated elements for lubricants for minimum 25 to 30% industrial applications.Potuz and Dassenoy[19] studied ultralow friction and wear behavior of single walled carbon nanotubes (SWCNTS). The addition of SWCNTs to lubricating oil has resulted in the drastic reduction of friction and wear. Yang et al. [20] have illustrated that well crystallized CNTs addition to lithium based grease is promising and effective in reducing friction and wear of tribological components. The work has employed two types of CNTs, which were synthesized by a floating catalyst method at different annealing temperatures to improve the lubricity, wear, and friction properties; the optimum percentage of the CNTs in the grease composites was 0.3wt.%, the friction coefficient was 0.125. As compared to the grease without the CNTs, the friction coefficient was reduced by 26%. Kobayashi et al. [21] has studied the effect of carbon nano-horn additives on grease lubrication to improve the wear, friction reduction, and EP properties. Used as lubricating additives have been investigated. They can greatly improve the AW and EP properties, reduce friction coefficient, and even retard the thermo-induced oxidation of lubricating oil/grease. Mohamed et al. [22] have depicted that CNTs addition to lithium grease has significantly improved anti-wear, anti-friction and extreme pressure characteristics of grease.

CNTs added lubricants performance has been widely studied. However, literature available on multiwalled-CNTs added lithium and calcium-based lubricants performance is limited. Hence, current work aims to investigate the effect of various weight percentages of CNTs on tribological behavior of CNTs added lithium and calcium grease. The synthesized CNTs was used in the study as an additives. The structural, wear and friction characteristics of CNTs added grease was studied.

II. EXPERIMENTAL METHOD

2.1 Material

The CNTs were blended with base grease comprised of lithium and calcium thickeners through mechanical blending as explained by Kobayashi et al. [21]. The percentage of CNTs was varied from 1 to 8 mass % in steps of 1%. Table 1 illustrates the chemical and physical properties of lubricant (as information provided by supplier).Structural characterization of CNTs was employed using high-resolution transmission electronic microscopy (HRTEM).

Table.1 Lubricant properties

Base oil	Poly-alpha-olefin(PAO)
Soap thickener	Lithium and calcium
Penetration (1/10mm at 25°C)	415
Dropping point (°C)	180
Viscosity of base oil to 40°C (mm^2 / s)	40

2.2 Experimentation

Tribological property of CNTs blended lithium and calcium grease was tested on a four ball test set up. The wear and friction test wasperformed according to ASTM D 2266 standard. The wear parameters viz. load, speed, duration of experimental run and operating temperature were considered at constant values as 392.26N, 600rpm, 60minute and 75°C respectively. The computed average Hertz stresses in the normal load of 392.26N was 0.392026 GPa. As per ASTM D2266 four ball testers receives maximum non-seizure loads. The used test balls (12.7mm, HRC60) aremade from steel (E-52200) with grade 25 extra polish. Each trial was repeated thrice and average reading was considered for the study. The friction coefficients were captured through data acquisition software connected to four-ball tester. The wear scar diameter (WSD) was measured using an optical microscope with precision of 0.15mm.

III. RESULT AND DISCUSSION

3.1 Structural Characterization of CNTs

The advanced multiwalled CNTs (MWCNTs) used were ultra-pure multi walled carbon nano tubes produced by catalytic carbon vapor deposition (CCVD) technique. X-ray diffraction (XRD) analysis of MWCNTs used in the study as shown in the fig.1. It depicts peak at 26 degree suggesting the presence of graphitic materials [15]. Fig.2 (a) and (b) illustrates the high-resolution transmission electron microscopy (HRTEM) images of Ru@ MWCNTs at 60,000 magnification and 1,20,000 magnification respectively. The average measure of CNTs were measured which lies in the range of 15nm approximately. It was noticed that, CNTs were long and thin in nature, which confirm its low aspect ratio. The low aspect ratio enables ease of transport of grease or lubricant during service with minimum accumulation effect.

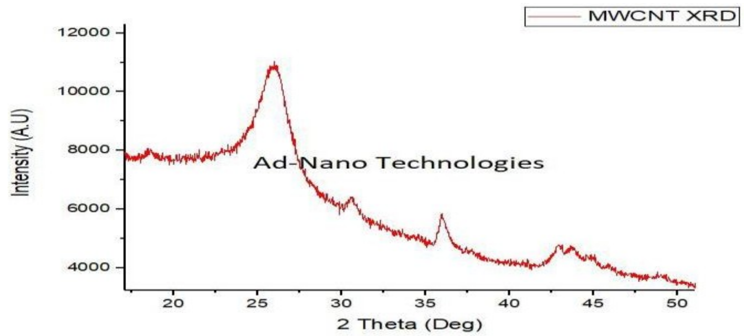

Fig.1. X-ray diffraction patterns of MWCNTs

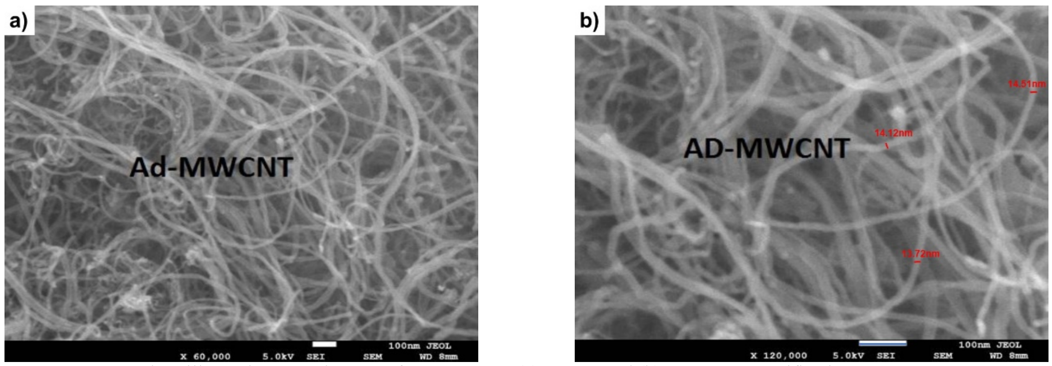

Fig.2 illustrating SEM images of MWCNTs at (a) 60,000 and (b) 1,20,000 magnification

3.2 Effect of CNTs addition on Tribological Behavior

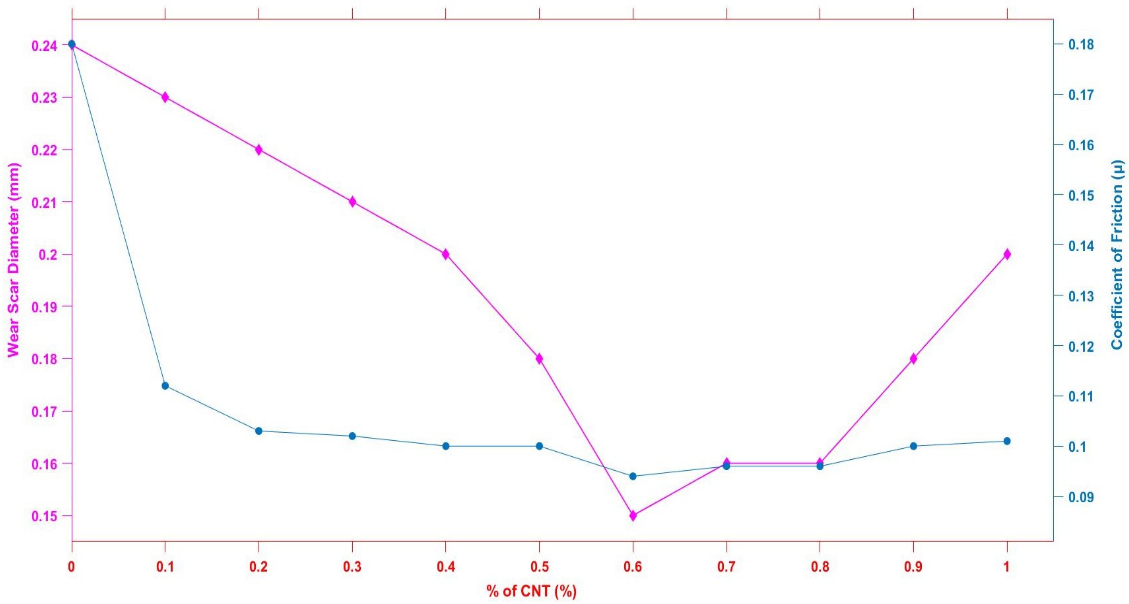

Fig. 3: Influence of % of CNTs on wear scar diameter

The influence of CNTs percentage on wear behavior of lithium and calcium lubricant was investigated. Further, CNTs percentage addition was varied from 0 to 1wt.%. Fig.3 illustrates the influence of CNTs addition percentage in lithium and calcium grease on wear scar diameter formed on steel balls. The plot suggests that increase in the CNTs percentage to 0.6wt.% lead to the decreased wear scar diameter owing to enhancement of wear resistance of lubricants. Conversely, addition of CNTs has improved the load carrying capacity of lubricant. However, beyond 0.6wt.% of CNTs addition has resulted in the increase of wear scar diameter suggesting declined wear resistance.

The effect of CNTs addition to lithium and calcium grease lubrication performance was studied as function of coefficient of friction (COF) (Fig. 3). The COF was found to decrease drastically with the addition of CNTs. Further, increase in the percentage of CNTs has resulted in the gradual decrease of COF. However, relatively slight increase in the COF was found when CNTs percentage was varied above 0.6wt.%. Thus, it can be inferred that 0.6wt.% addition of CNTs to lithium and calcium grease is optimal and lucrative in engineering applications. It is attributed to the presence of synergistic action of CNTs and grease studied aids in reducing COF and enhancing wear resistance. When CNTs addition was increased above 0.6wt.% perhaps has caused detrimental effect on synergistic performance of CNTs/grease. It might be due to the fact that, increase in CNT above 0.6wt.% might has increased the number of CNTs entrapped between contacting surfaces. It has resulted in increased sonication, which lead to decline of adhesion, uniform dispersion and ease of flow characteristics. Thus, antagonistic phenomenon was resulted causing detrimental effect. However, the coefficient of friction and wear resistance of 1wt.% CNT added grease was relative better compared to neat grease

3.3 Worn Surface Analysis

The CNTs added lithium and calcium grease was subjected to lubrication mechanism between steel balls. Subsequently, variation of CNTs percent to lithium and calcium grease on its tribological behavior was investigated. Worn surface analysis of steel balls was employed through their scanning electron microscope (SEM) images. Fig. 4(a), (b) (c) and (d) illustrates the SEM images of worn surface of tested steel balls when they were in lubrication of neat grease, 0.4wt.%, 0.5wt.% and 0.6wt.% grease respectively. The coarse and severely rubbed marks were evident on the surface lubricated with neat grease (fig. 4(a)). While, fig. 4(b) illustrates the reduced diameter of scar caused due to rubbing on the surface lubricated with 0.4wt.% of CNTs added grease. Furthermore, scuffed surface was found to be diminished in fig. 4(c) and minimum scuffing was evident as shown in fig. 4(d). The visual examination of balls surface indicates that initial scratching, polishing and pullout of asperities has disappeared. The existence of transfer layer formed during lubrication was noticed. The transfer layer acting as tribo-film was generated during lubricating acting has endured the load considerably perhaps due to the addition of CNTs into grease. Fig. 5 and fig. 6 depicts the EDS analysis of worn surface lubricated with 0.1wt.% and 0.6wt.% CNTs added grease respectively. It demonstrates the presence of C element on the surface with atomic 5.89% and 12.69% corresponding to 0.1% and 0.6% CNTs added grease lubricated surface. Thus, it implies the occurrence of possible diffusion and deposition of carbon atoms. The percentage of C also has increased owing to formation of tribo-layer was mainly due to CNTs addition.Therefore, presence of C element substantiates that it is sufficient to shape a lubricating tribo-film comprising carbon rich layer and inhibit direct steel-to-steel contact during rubbing action [22].

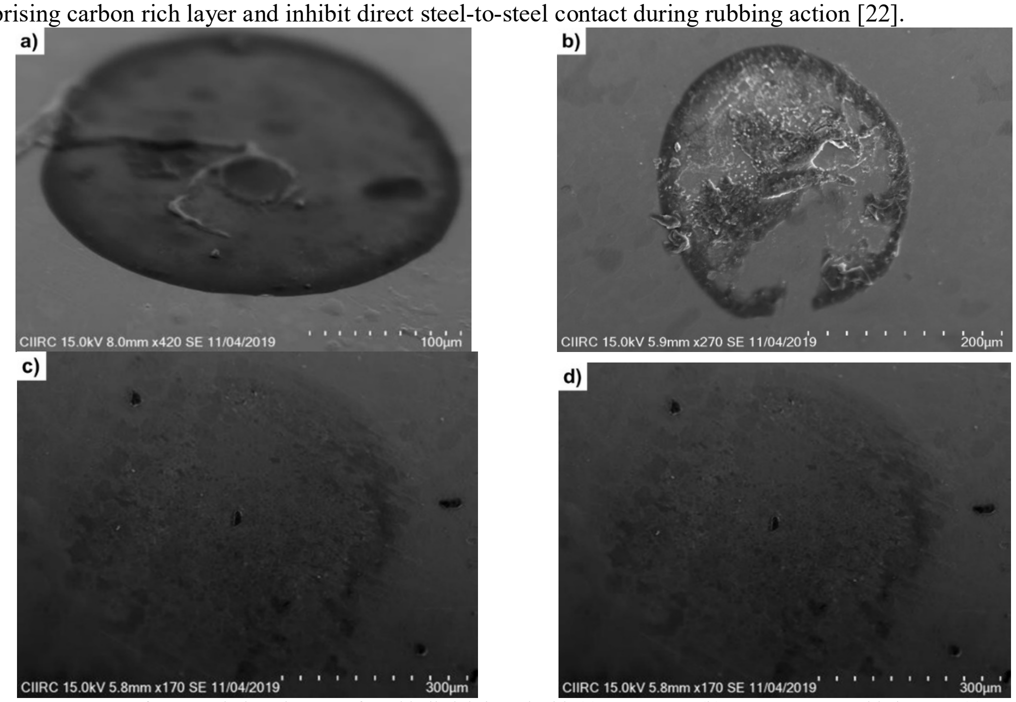

Fig. 4 SEM worn surface morphology images of steel balls lubricated with (a) neat grease (b) 0.4wt.% CNTs added grease (c) 0.5wt.% CNTs added grease (d) 0.6wt.% CNTs added grease

The carbon-rich layer was enhanced in 0.6wt.% CNTs added grease compared to 0.1wt.% CNTs addition.Consequently, it confirms that 0.5wt.% and 0.6wt.% CNTs addition to grease has enhanced further lubrication ability of grease. In addition, 0.6wt.% CNTs added grease lubricated surface exhibited smooth surface with reduced scar size. Also, 0.6wt.% CNTs addition to lithium and calcium grease was optimum to achieve its better anti-wear and anti-friction characteristics, which is in good agreement with earlier reported studied [19,22].

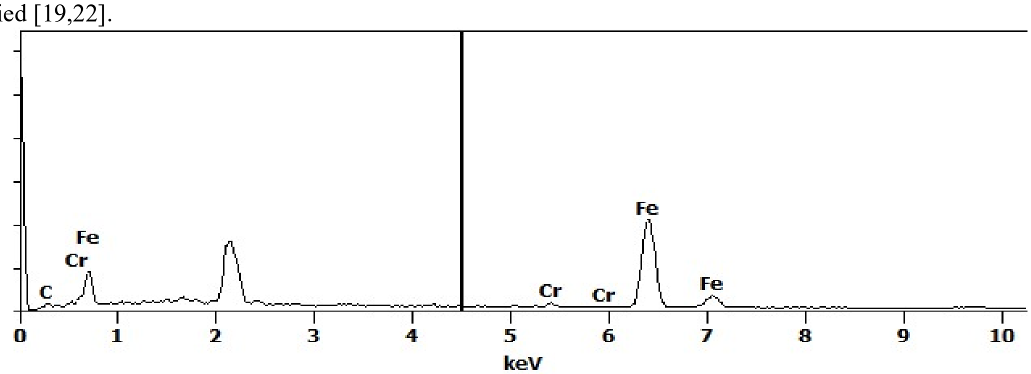

Fig. 5 EDS analysis of steel ball lubricated with 0.1wt.% CNTs added grease

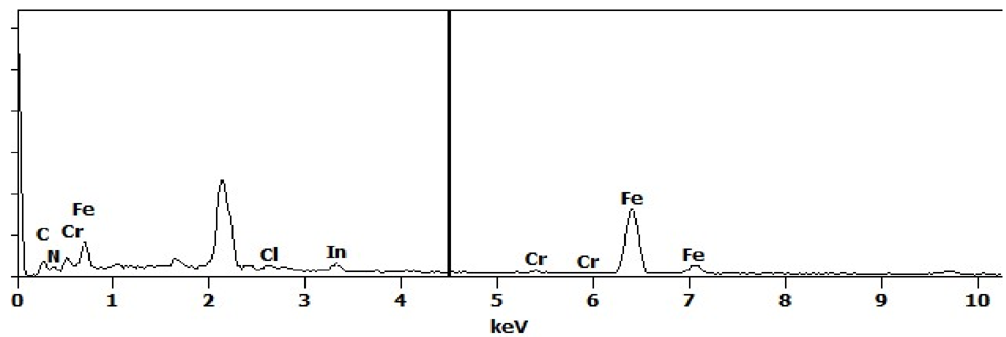

Fig. 6 EDS analysis of steel ball lubricated with 0.6wt.% CNTs added grease

IV. CONCLUSIONS

The investigationwas carried to study the influence of CNTsaddition, and also its % variation on the tribological behavior of lithium and calcium grease. Based on the obtained results following conclusions were drawn.

- The CNTs addition to lithium and calcium grease has enhanced its wear resistance and reduced coefficient of friction.
- The variation of % addition of CNTs has decreased the wear loss and COF of lubricated contact steel balls. Besides, declining trend was observed till 0.6wt.% of CNTs beyond which it has caused in the increase of wear and COF. However, COF and wear resistance noticedstill relatively better at 1wt.% of CNTs compared to neat grease.
- The improved tribological characteristics was attributed to the formation of lubricating tribo-film comprising carbon atoms between contacting surfaces during rubbing. The carbon atoms presence was confirmed through EDS analysis of 0.6wt.% CNTs lubricated surface. Thus, tribo-film inhibited the direct steel-to-steel contact and contributed to enhance its anti-wear and anti-friction characteristics.
- SEM analysis of worn surface indicated that, coarse and severely rubbed marks with larger scar diameter on the neat grease lubricated surface. The scuffing marks was found to be decreased with reduced scar diameter on lubricated surfaces with CNTsadded grease. In addition, least scuffed markwas observed on surface lubricated with 0.6wt.% CNTs added grease validating superior wear resistance.

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