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Graphite Nanoplatelets Based Advance Lubrication Agent with Engine Oil for Hard Thermal Spray Coating

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Abstract

The target of this article is to explore the fundamental tribological behavior of graphite nanoplatelets, evaluate its performance as a self-lubricating material. The wear and tribological behavior of graphite nanoplatelets with engine oil under sliding conditions has hardly been investigated. This paper is concerned with the importance of Nano lubricant (Graphene Nanoplatelets + Engine Oil) due to the promising application of friction and wears reduction.

Hence, first of all HVOF (High Velocity Oxy-Fuel) WC-10Co-4Cr coating was deposited on titanium substrate. Then, the dispersion stability of GrNP (Graphite nanoplatelets) in lubricants is studied immediately after sonication and after 7 days of sonication.

A ball on disk tribometer has been used to verify the friction and wear reduction due to graphite nanoplatelets on thermal spray coating under two conditions i.e. dry sliding (without lubricant) and engine oil added graphite nanoplatelets (GrNP), at 0.3m/s sliding speed, 20m sliding distance, 30N Load for 15min.

The Coefficient of friction (COF) of WC-10Co-4Cr coating is in the range of 0.01-0.18. Engine oil with optimized concentration of GrNP i.e.0.21 wt.% showed enhanced friction compared to WC-CoCr coating (without lubricants). The friction coefficient reduced by 34%, (Engine Oil), and approx. 83 % for (Engine oil +GrNP). Similarly, GrNP +lubricants greatly reduced the wear volume loss to 84% (Engine oil +GrNP). A significant decrease in wear rate from 1.0E-2 3/N. m to 1.0E-6 3/N. m is also reported with GrNP+lubricants compared to WC-CoCr coating (without lubricants).

Key Words: Lubrication; Tribology; Wear; Graphite Nanoplatelets; Engine oil; Friction co-efficient

Introduction

The application of the engineered surfaces depends on compatibility to the counter surfaces. Minimizing machine failure, energy loss, friction and mechanical wear failure by lubrication and to enhance service life is a challenging situation for researchers and scientists. Reduction in wear and friction mainly depends on conditions such as, relative motion between the surfaces, sliding speed, normal load, vibration, temperature and lubrication etc. [1].

A surface is itself a defect and regardless of its ultimate finished form it always has depressions, valleys and ridges [1]. So, lubrication with a better nano additive provides the effective mechanical functioning along with improved energy efficiency and durability. Severe tribological conditions exist in military combat operations, aircraft, automotive industries, an efficient lubricant is demanded always [2].

Control over wear and friction losses using conventional lubricants was a long-ago practice in many applications[3]. But the conventional lubricants are unsatisfactory for the use of commercial applications because of pungent odor, bad thermal stability, and corrosion as it contains sulphur and phosphor[4]. Therefore, quest for nano particles as new kind of additive for lubrication for systemic control over wear and friction has significantly increased from the past few years.[5] Certain nanoparticles such as, metal sulfides,[6] metal oxides,[7] carbon materials, organic material etc. have been investigated as anti-wear agents. Studies have shown the superior lubrication and tribological properties of nano additives than conventional lubricant solid additive. Advantage of using nano additive lubricant over conventional lubricants as anti-wear agents are size and shape effect, enter contact asperities, better chemical reactivity, better protective film, third body effects, limited tribo-chemical reactions etc. These nano additives are added in a low concentration to the base oil (synthetic oil, mineral oil) and brings improvement in the tribological characteristics and presenting good wear and friction reduction effects. Several anti -wear and frictional mechanisms are reported in the previous papers using titanium dioxide nanoparticles,[8] graphene, graphene flakes, tungsten disulphide etc.

Nano carbon materials have received great attention by tribology researcher due to high load carrying capacity, high chemical stability, strong intramolecular bond, and low surface energy. For practical applications, graphene nanoplatelets have been the focus of interest due to its remarkable properties and unique structure. However, few studies on graphene nanoplatelets have been reported so far. Graphene nanoplatelets have been used for better lubricity and wear resistance properties when used as an oil additive. Huang et al.[9] investigated the frictional behavior and anti-wear property of paraffin oil when graphite nano sheets were added. Lin et al.[10] investigated the tribological properties of graphite nanosheets as an oil additive. Weak intermolecular forces (Vander wall forces) exists between the layers, that enhanced that anti-wear properties. Better friction properties of oleic acid is obtained when graphene sheets were dispersed in lubricant oil.[11] Prasad et al.[12] investigated sliding wear behavior of gray cast iron at different loads using mineral oil additive graphite nanoparticles. At low concentration (5wt%) decrease in wear rate is noticed, while when the concentration is increased to 10wt% the wear rate significantly decreased at high loads. Hwang et al. [13] found the improvement in friction reducing and antiwear property of mineral oil when graphite nanoparticles were added. These spherical shaped nanoparticles prevented direct contact between the sliding surfaces. The literature review shows that less work has been done on improving the tribological properties of HVOF sprayed WC-10Cr-4Co coatings with lubrications added graphene nanoplatelets.

Therefore, the objective of this paper is to investigate the tribological properties of graphene nanoplatelets as nano additive and reveal the lubrication mechanism of graphite nanoplatelets.

1. EXPERIMENTAL WORK

1.1. Sample Formulation (Lubrication samples)

Graphite nanoplatelets-GrNP (Purity>99.5wt%, <22 layer, thickness: 4-18nm, Size 5-9µm) purchased from Chengdu Organic Chemicals Co. Ltd., Chinese Academy of Sciences were used throughout the experimental work. API 20W-50 CH-4, SL engine oil was purchased from Guangdong Litten Lubricants Co. Ltd. China and used as a baseline lubricant in this experiment. The lubrication samples were prepared with as purchased GrNP using ultrasonication, leading to samples of desired percentage of 0.21wt% concentration. The lubrication samples with concentration of 0.21wt% were prepared at ambient temperature in 20W-50 engine oil by dispersing GrNP in 0.0525 g/L of engine oil. Then mixed ultrasonically (40% amplitude, 2s pulse, 50Hz frequency) for 35min 20W-50 engine oil +GrNP to make sure that GrNP is well dispersed. The lubricant (20W-50 engine oil) without GrNP was used as a baseline in this work.

The properties of graphite nanoplatelets and properties of engine oil are given in Tables 1.1 and 1.2.

Graphite nanoplatelets	Properties
Average size (µm)	43594
Purity (%)	99.5
Thickness (nm)	43573
Layers	<22
Density (g/cm^3)	0.5
PH Value (30°C)	7.05 -7.60

Table 1.1: Properties of graphite nanoplatelets (Chengdu Organic Chemicals Co. Ltd., Chinese (Academy of Sciences)

Properties	API CH-4/ 228.1, SL
SAE Viscosity grade	20W-50
Kinematic Viscosity (@40˚C)	165
Kinematic Viscosity (@100 ˚C)	18
Dynamic Viscosity (@-15 ˚C)	8400
Viscosity Index	134
Flash Point -˚C	245
Pour point -˚C	-27
Color	L 4.0
Density (@15 ˚C) kg/l	0.889
Zinc (wt. %)	0.115

Table 1.2: Properties of Engine oil (Guangdong Litten Lubricants Co. Ltd. China)

1.2. HVOF Sprayed WC-Co-Cr Coating Sample

Cermet powder with the particle size(-45+11μ) was purchased from Guangzhou Sanxin Metals S&T from china, was used for HVOF sprayed WC-Co-Cr coatings. Titanium substrate plates from Guangzhou Sanxin Metals S&T from china were sand blasted to enhance the mechanical interlocking of powder to the substrate. Coating was deposited with HVOF Sulzer Metco (Institute of Space Technology, Islamabad, Pakistan-IST) with water cooled diamond jet gun DJ8A hardware 2600, with high pressure, high velocity flame spraying torch which operates at reduced temperature (Figure 1.1). The fuel used throughout the experiment was hydrogen.

Figure 1.1: HVOF Sulzer Metco with water cooled diamond jet gun DJ8A hardware 2600 (IST)

The spraying parameters and basic composition of a disk are given in Tables 1.3 and 1.4

Flow rate	**10.84L/min**
Powder feed	5.1rpm
Spraying distance	300mm
Flame temperature	2500K

Table 1.3: Spraying parameters

Tribological test

A pin-on-disk tribometer 10.2. friction and wear tester, installed in Institute of Space Technology, Islamabad, Pakistan (IST) was used to study the tribological properties of lubricants effect on HVOF sprayed WC-Co-Cr coating.

The pin-on-disk tests were performed against a 3mm steel as a counter-part acting in relative to the loaded coated HVOF sprayed WC-10Co-4Cr disk. Before wear test the surface of the disk was ground with 800 and 1000 mesh sandpaper, respectively.

The sliding wear resistance of the samples were tested under dry condition and with graphene nanoplatelets added lubrications. The friction co-efficient, and wear rate was recorded automatically during the test, with the built-in channels of tribometer. The topographical images of the samples were observed using scanning electron microscope. The experimental conditions are given in Table 1.4.

Figure 1.2: Pin-on-disk tribometer 10.2 (IST)

Figure 1.3: Diagram showing substrate holder and pin (IST)

Parameters	Values
Normal load (N)	30
Abrasive material	Steel pin
Test duration	15 min
Sliding velocity	0.3m/s
Temperature	Ambient
Lubricants	Engine oil modified with graphite nanoplatelets
Sliding distance	20m
Type of motion	Relative
Speed	200 rpm

Table 1.4 Experimental conditions

2. Results

2.1. Graphite Nano platelets (GrNP) and HVOF WC-10Co-4Cr Coating

Characterization

Figure 2.1 and 2.3 showed the SEM images of HVOF WC-10Co-4Cr coating and graphite nanoplatelets GrNP. The ‘flake-like’ morphology of graphite nanoplatelets (GrNP) can be seen in figure 2.3. The typical EDS analysis of WC-10Co-4Cr coating is shown in figure 2.2.

2.2. Structure and Dispersion Stability of GrNP

The as-prepared GrNP was dispersed in 20W-50 SL engine oil to a nominal concentration of 0.21wt% with the aid of ultrasonication, and the dispersion were allowed for seven days. Figure2.4 shows the dispersions immediately after sonication (20W-50 engine oil+ GrNP). To identify the degree of dispersion and sedimentation, pictures were again taken after 7 days (figure 2.5). Just after sonication, GrNP showed good dispersion with the engine oil. The multilayered GrNP showed good dispersion without surface modification or adding any surfactants. Moreover, it is evident that carbon-based materials stability in an aqueous medium is highly dependent on the hydrophilic functional group, which interacts with the repulsive force (columbic) and hydrogen bonding. Therefore, for GrNP, the defect on its edges contain unfilled carbon bonds that assist in providing favorable sites for oxidative reaction. Thus yielding more hydrophilic structures which comprises of various functional groups (water based), such as ethers, carboxyl and hydroxyls [14].Thus its solubility is size-dependent since more functional groups exist in larger sheets, which contains structural defects[15].

After 7 days, the figure 2.5 showed some sedimentation and partially settling of GrNP in engine oil. The rate of settling is highly dependent on the density difference between the GrNP and the engine oil and viscosity of engine oil etc. The interaction potential and forces between the layers, attracts each other and agglomeration occurs.

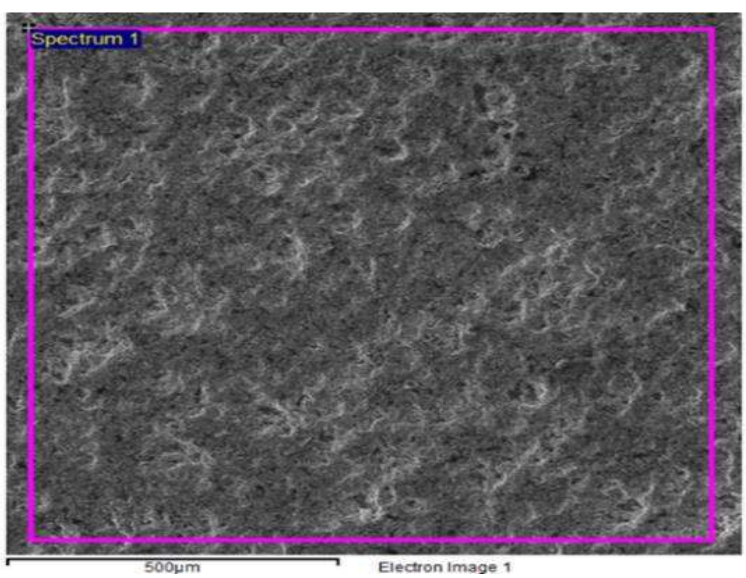

Figure 2.1: SEM image of as sprayed HVOF coating WC-10Co-4Cr

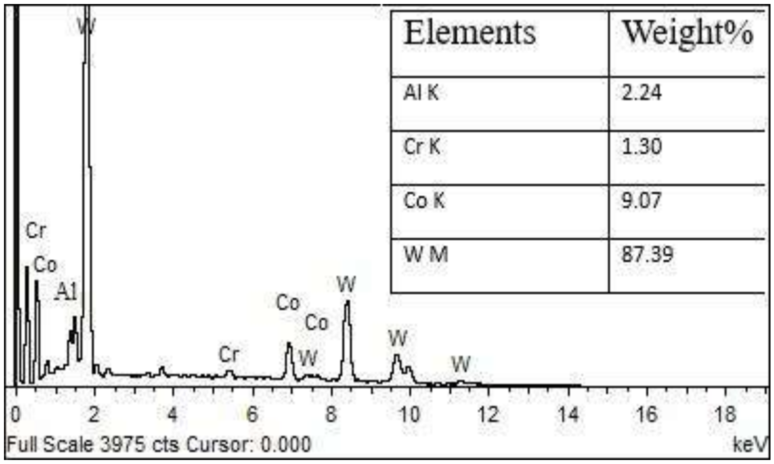

Figure 2.2: EDS of HVOF coating WC-10Co-4Cr

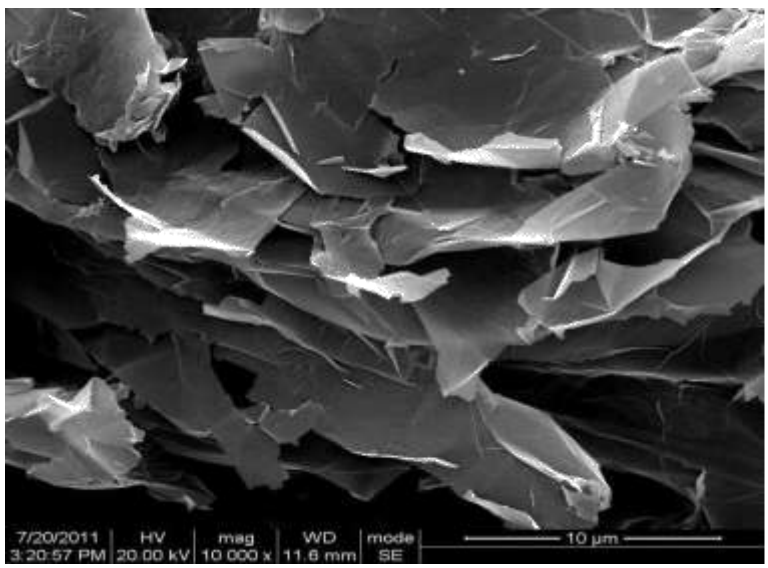

Figure 2.3: SEM of graphite nanoplatelets (Chengdu Organic Chemicals Co. Ltd., Chinese Academy of Sciences)

Figure 2.4: a) Engine oil+ graphite nanoplatelets (GrNP), b) Engine oil (without graphene nanoplatelets (GrNP) - (Day 1)

Figure 2.5: a) Engine oil+ graphite nanoplatelets (GrNP), b) Engine oil (without graphene nanoplatelets (GrNP)- (Day 7)

2.3. Tribological Characteristics and Characterization

2.3.1. Influence of graphite nanoplatelets on friction-reducing capability of lubricants

Figure 2.6 shows the variation of friction coefficient with friction time of different conditions. It can be seen from Figure 2.6 that, under dry friction condition, with the increase of friction time, friction coefficient increased firstly and then decreased. The increase of COF is due to the continuous generation of wear debris in the form of hard particles entrapped between the worn surfaces. Plastic deformation process increases the hardness of the debris particles and they scrape the surrounding material. COF then reached to 0.02, suggested that, thick oxide layer formed on the surface due to oxidation. The oxide formed on the surface adheres to the surface as thick layer and provide protection against the sliding asperity.

Under lubrication conditions the behavior of engine oil on coating was different while following the same trend i.e. increased firstly with friction time and then reached a stable state value. Test performed with base oil without GrNP showed the high COF (0.07), suggesting that graphite nanoplatelets is most likely the reason for the friction reduction. This can also be the suggested that on contact with the counter body local surface temperature rise occurred due to low thermal conductivity that reduces the viscosity of oil to some extent as compared to GrNP+Oil. The fairly stable COF attained is due to the graphitic layers entrapped in the contact area is high and continuous lubricating film comprising of graphene was formed on the surface. Low concentration of GrNP in solution forms a protective tribofilm, resulting in low and stable COF.

2.3.2. Effect of rotation speed on friction

To study the tribological properties of the tested lubricant along different lubrication regimes, the experimental approach proposed by Kovalchenko et al. (2005) [16] is adopted for the current study.

The friction test begins with an increasing rotating speed until 250rpm for 300s. The shape of graph shows a transition behavior from dry condition-mixed lubrication regime-elastohydrodynamic lubrication regime (EHL)-hydrodynamic lubrication regime (HL-full film lubrication) for increasing speed [17].

At lower rotational speed (0-4rpm), specially at transition point B in figure 2.7, the lubrication 20W-50 SL engine oil and GrNP+Oil is EH. At higher rotating speed lubricant showed the transition to HL i.e. full film lubrication regime, where the friction is determined by the viscosity of laminated GrNP. COF of GrNP+Oil is lowered than 20W-50 SL base oil. This might be attributed to the exposure of GrNP lamellar layers under the applied load. A positive effect of GrNP on lowering the friction coefficient is specially observed in EHL regime where the viscous stress is prevalent. Moreover, increase in COF in HL regime shows that after a transition from EHL to HL, lubrication suffer from wear and some particles worn out from the surface but later form a uniform tribofilm and reduces the energy loss in mechanical lubrication. These tests confirmed that using GrNP lubricants enhances the friction reduction capability in mixed, EHL and HL regime as reported in [17].

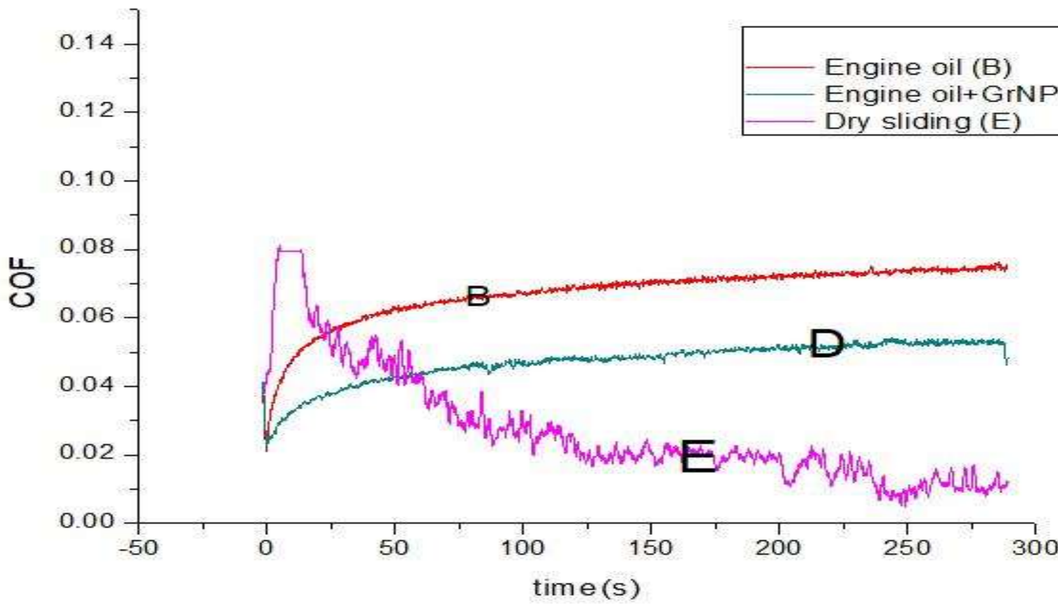

Figure. 2.6: Variation of friction coefficient with friction time for various lubrication conditions i.e. engine oil with graphene nanoplatelets (GrNP) and engine oil (without graphene nanoplatelets) – and dry sliding: Where "COF" means Coefficient of friction

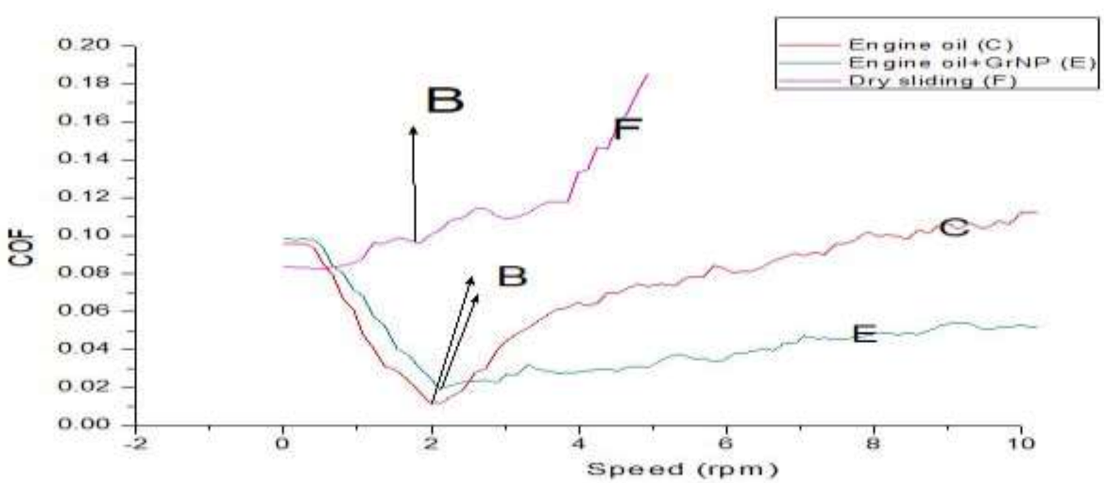

Figure 2.7: Effect of rotational speed on friction behavior of engine oil with graphene nanoplatelets (GrNP) and engine oil (without graphene nanoplatelets) and dry sliding at RT -*Where "COF" means Coefficient of friction*

2.3.3. **Influence of graphite nanoplatelets on wear-reduction capability of lubricants**

Figure 2.8 shows the variation of wear rate with sliding distance for various conditions. Wear rate increased initially and then decreases with increasing sliding distance. It can be inferred that lubrications with GrNP showed less wear rates as compared to without lubrication due to their thin laminated structure which allows the GrNP to form self-assembled tribofilm and reduces the direct contact between the two surfaces. Graphene stacking piles takes place and Vander wall forces between different layers helps in the slipping process that lowers the wear rate. Moreover 0.21wt% concentration also effects in lowering wear mechanism [18].

2.3.4. **Observation of wear tracks**

Microscopic examination of the wear track showed the presence of debris within the wear track. Debris are of whitish color regardless of the lubricant used. Debris are of two kinds flakes or platelets (20W-50 engine oil+ GrNP) and finer particles (20W-50 engine oil).

The SEM of WC-10Co-4Cr coating after dry sliding test (without lubrication) shows the spalling of Co/Cr binder phase. This is due to the counter steel contact. The presence of Fe and Cr showed the strong contact and extrusion between the sliding bodies. From the SEM images, it can be suggested that groove marks are filled with the graphite nanoplatelets provided a decrease in COF and wear rate in 20W-50 engine oil+ GrNP. These accumulated platelets act as a load carrying entities and significantly affect the wear rate. In case of engine oil, one possible explanation for this behavior may be due to the agglomeration and accumulation of GrNP occurs in the contact area. This reduces the lubricating effect of solution and micro plowing and micro cracking can be seen.[19]. Figure 2.13 shows spalling, deformation and cracks on the wear surface of the coating under dry condition. This indicates that coating suffers from severe damage without lubrication at 30N load.

The examination of the worn surface has thus allowed the detection of the presence of platelets in either form i.e. particle or agglomerated debris. The debris contributed to the wear process through the following mechanism.

1. Micro-cracking and micro-plowing along the sliding contact (20W-50 engine oil)
2. Abrasion by accumulated platelets and flakes of GrNP and fine debris circulating along the sliding contact (20W-50 engine oil+ GrNP).

The EDS spectrum in figure 2.2 shows the presence of trace amount of Al, which is due to the impurities present on the surface. The lubrication mechanism of lubricants can be confirmed by the results of SEM and EDS. Owing to small size and high surface energy of GrNP, they could stably form a tribofilm on the surface of HVOF coating. The EDS spectra for 20W-50 engine oil+ GrNP revealed a carbon characteristic peak, which indicates that GrNP is transferred to the surface to lubricate the friction pairs. The presence of Fe and chromium is due to the steel contact base oil. While the oxygen was due to the oxide layer formed in engine oil.

2.3.5. **Variation of wear volume with respect to Sliding distance**

The figure 2.15 shows that the wear volume value is higher in case of engine oil with respect to sliding distance. Figure 2.15 shows that GrNP greatly reduced the contact between the sliding bodies and less penetration takes place, so wear volume is just related to surface lubricants. Engine oil showed the higher penetration and wear volume due to lack of GrNP tribofilm, while GrNP+oil showed less wear volume and penetration depth.

Moreover, figure 2.15 further reveal that GrNP+oil have the higher peak intensity initially and reaches up to the peak value of $4.0\times 10^{-7\ 3}$. (GrNP+oil). While for engine oil the peak value comes at $5.0\times 10^{-7\ 3}$. The lubrication film of engine oil with GrNP showed good wear resistance due to graphite nanoplatelets.

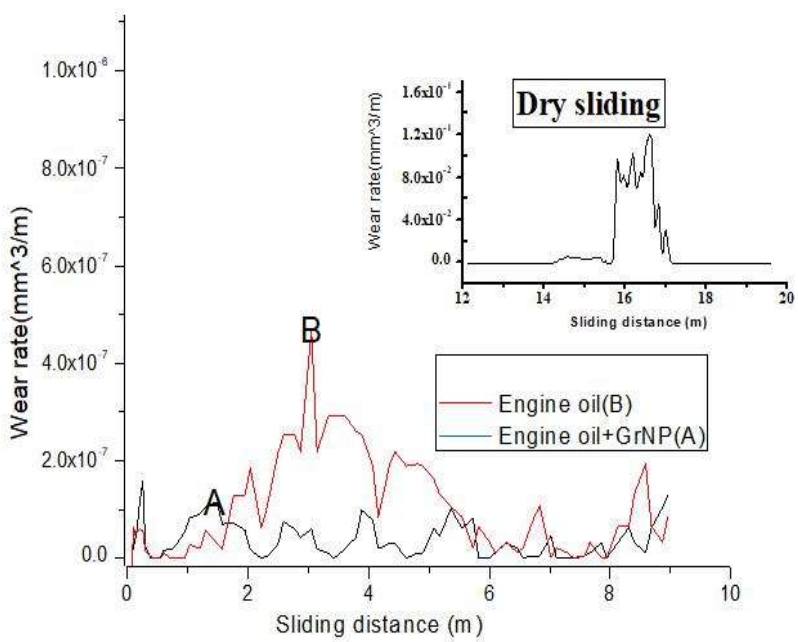

Figure 2.8. Variation of wear rate with sliding distance for various lubrication conditions i.e. engine oil with graphene nanoplatelets (GrNP) and engine oil (without graphene nanoplatelets) and dry sliding

Figure 2.9: SEM image of graphite nanoplatelets (GrNP)+ engine oil dispersed on WC-10Co-4Cr coating

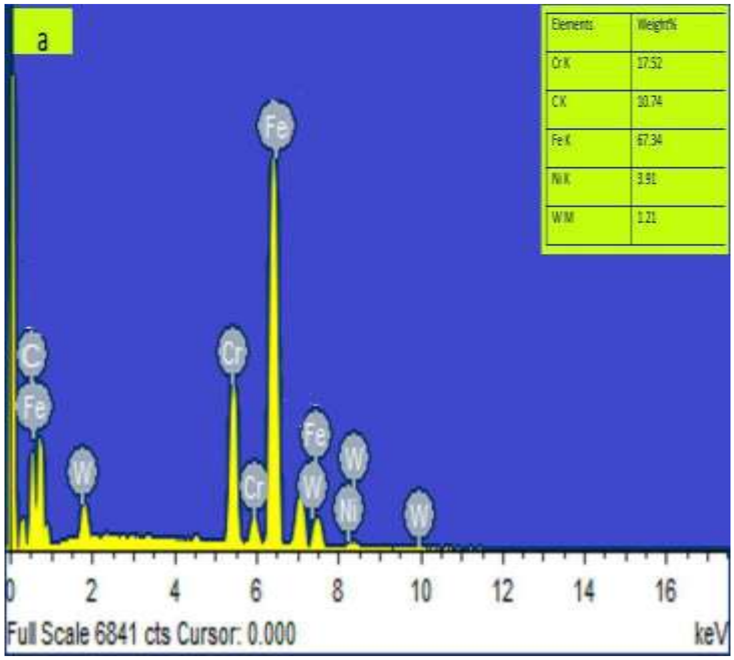

Figure 2.10: EDS of graphite nanoplatelets (GrNP)+ engine oil dispersed on WC-10Co-4Cr coating (area A in fig 2.10)

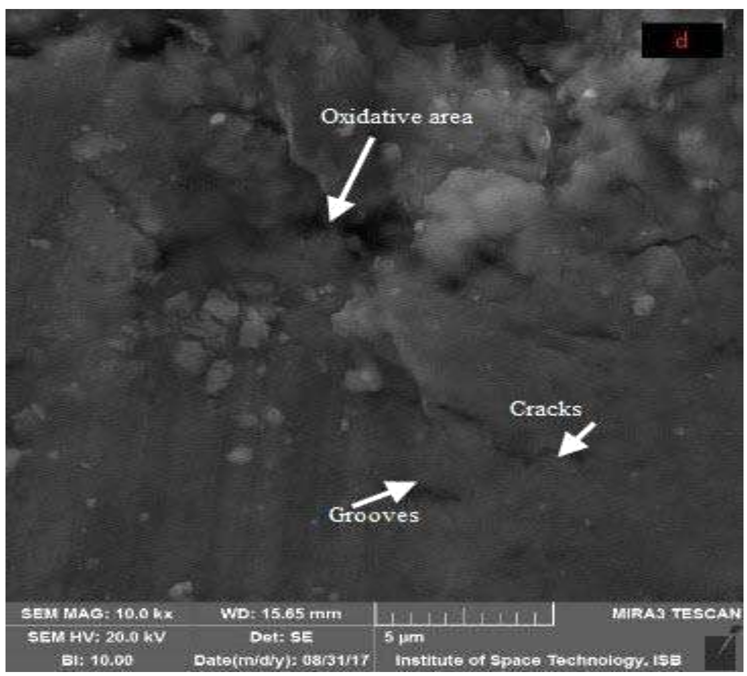

Figure 2.11: SEM image of engine oil (without graphite nanoplatelets) dispersed on WC-10Co-4Cr coating

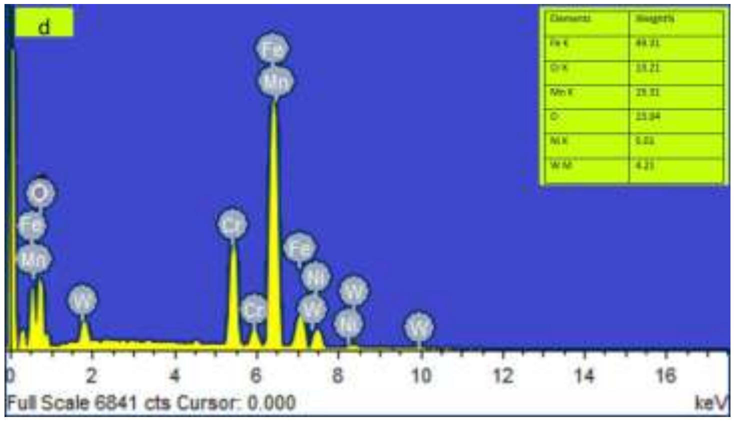

Figure 2.12: EDS of engine oil (without graphite nanoplatelets) dispersed on WC-10Co-4Cr coating (area D in fig 2.12)

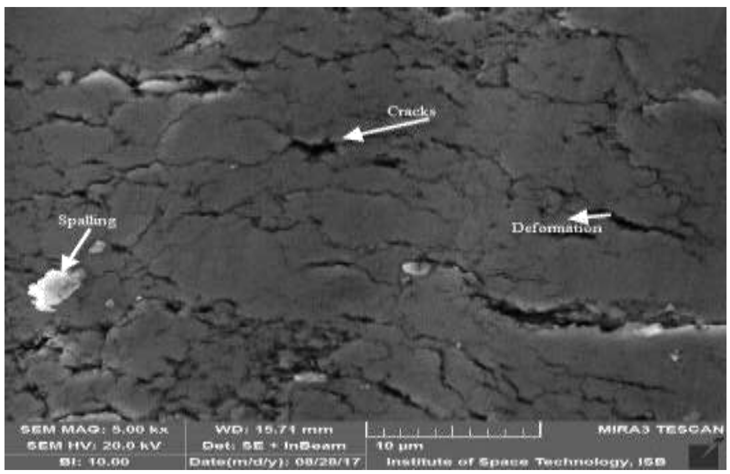

Figure 2.13: SEM image of WC-10Co-4Cr coating after dry sliding test (without lubrication)

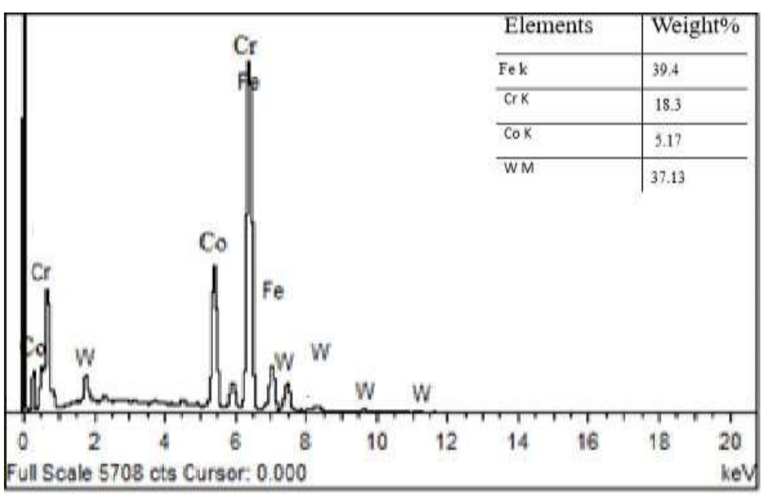

Figure 2.14: EDS of WC-10Co-4Cr coating after dry sliding test (without lubrication)

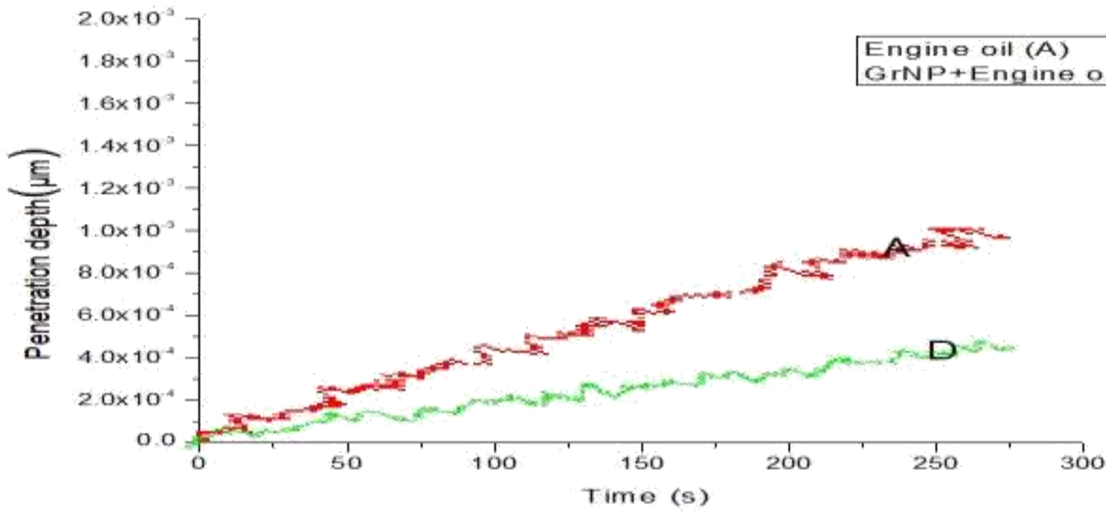

Figure 2.15: Wear volume under different lubrication conditions i.e. engine oil with graphite nanoplatelets (GrNP) and base oil (engine oil without graphite nanoplatelets) and dry sliding

2.3.6. **Penetration depth with respect to time**

Fig 2.16 shows the penetration depth with respect to time under different lubrication conditions. The graph revealed that penetration depth increases with respect to time in case of engine oil and showed the valued of 13µm approx. This behavior can be easily understood with respect to wear volume and wear rate. As the wear rate increases the penetration depth increases.

Penetration depth values for engine oil +GrNP is considerably less. Its values range in 4-6 µm approx. showing that wear rate is less. So less penetration with respect to time can be seen.

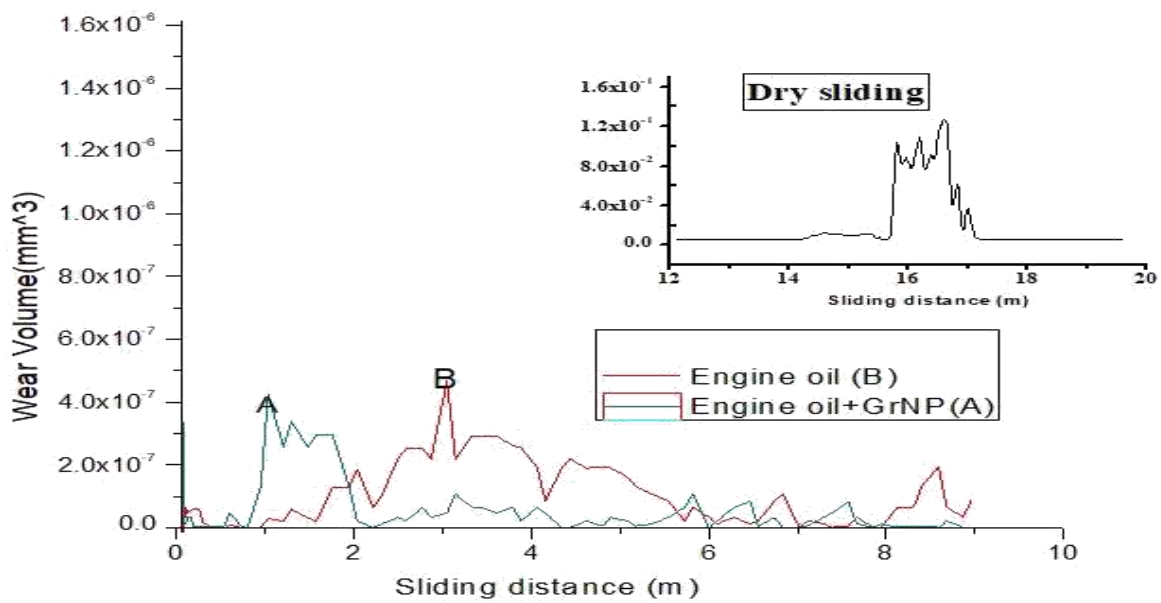

Figure 2.16: Penetration depth under different lubrication conditions i.e. engine oil with graphene nanoplatelets (GrNP) and base oil (engine oil without graphene nanoplatelets)

3. Conclusion

A series of experiments were conducted to test the wear and friction behavior of HVOF sprayed WC-10Co-4Cr coatings under dry and two lubricated conditions. Wear of the coating was greatly reduced (few orders of magnitude) with reduction in friction coefficient by the presence of graphite nanoplatelets at the tribological surface as compared to dry condition test.

1. The best antiwear result has been observed in hydrodynamic, mixed and Elasto-hydrodynamic conditions.
2. The good friction and antiwear properties of graphite nanoplatelets is due to their multiple layered structure which offers least contact between the two sliding bodies.

4 Future work

Future work includes

Should develop better dispersion methods along with different dispersant for GrNP dispersion in base oil.

Search for optimal concentration of GrNP in different bases, for better viscosity and better anti-wear property.

Search for more nano-lubricating agents which have low surface tension and incorporates in the pores of WC-Co-Cr coating for better wear.

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